Bruno Zaslick

The Electrochemical Society . . .

120th Meeting

Detroit
October 1, 2, 3, 4, and 5, 1961

LOCAL COMMITTEE

General Chairman

Manuel Ben

Vice-General Chairman

Seward Beacom

Registration

John Griffin

Finance

Sidney Selis

Entertainment

Sam Piken

Plant Tours and Transportation

Frank Passal

Publicity

James Hoare

Hotel Arrangements

Robert Trees

Ladies' Committee

Mrs. Ann Piken

Detroit Meeting

THE ELECTROCHEMICAL SOCIETY

October 1, 2, 3, 4, and 5, 1961

Sunday through Thursday

CONDENSED TECHNICAL PROGRAM

Monday, October 2, 1961

Battery—Corrosion (Abstracts 32-44) 9:00 A. M.-12:15-P.M.; 2:00-5:10 P.M. ~~(Tuller Room~~, Hotel Tuller, ~~14th floor)~~ Arabian Ballroom
Electrodeposition (Abstracts 69-77) 9:00 A.M.-12:05 P.M.; 2:00-4:45 P.M. (Wayne Room, Ballroom floor, Statler-Hilton Hotel)
Electronics-Semiconductors (Abstracts 113-130) 9:00 A.M.-12 M.; 1:30-5:00 P. M. (Grand Ballroom, Statler-Hilton Hotel)
Electro-Organic (Abstracts 158-165) 9:00 A. M.-12:00 M.; 2:00-5:30 P.M. (English Room, Ballroom floor, Statler-Hilton Hotel)
Electrothermics and Metallurgy (Abstracts 179-186) 9:00 A.M.-12 M.; 2:00-5:00 P. M. (Bagley Room, Ballroom Floor, Statler-Hilton Hotel)

Tuesday, October 3, 1961

Battery (Abstracts 1-14) 9:00 A.M.-12:00 M.; 2:00-4:55 P.M. (Tuller Room, Hotel Tuller, 14th floor) Arabian Ballroom
Corrosion (Abstracts 45-56) 9:00-11:40 A.M.; 2:00-4:40 P.M. (Michigan Room, Ballroom floor, Statler-Hilton Hotel)
Electrodeposition (Abstracts 79-90) 9:00 A. M.-12:00 M.; 2:00-5:00 P.M. (Wayne Room, Ballroom floor, Statler-Hilton Hotel)
Electronics-Semiconductors (Abstracts 131-143) 9:00 A.M.-12:00 M.; 2:10-5:00 P.M. (Grand Ballroom, Statler-Hilton Hotel)
Electro-Organic (Abstracts 166-171) 9:00 A.M.-12:10 P.M.; 2:00-5:00 P.M. (English Room, Ballroom floor, Statler-Hilton Hotel)
Electrothermics and Metallurgy (Abstracts 187-194) 9:00-11:20 A.M.; 2:00-4:30 P.M. (Bagley Room, Ballroom floor, Statler-Hilton Hotel)

Wednesday, October 4, 1961

Battery (Abstracts 15-25) 9:00 A.M.-12:00 M.; 2:00-5:00 P.M. (Wayne Room, Ballroom floor, Statler-Hilton Hotel)
Corrosion (Abstracts 57-68) 9:00-11:15 A.M.; 2:00-4:40 P.M. (Michigan Room, Ballroom floor, Statler-Hilton Hotel)
Electrodeposition (Abstracts 91-106) 9:00 A.M.-12:00 M. (Tuller Room, Hotel Tuller, 14th floor); 9:00 A.M.-12:00 M.; 2:00-5:00 P.M. (Bagley Room, Ballroom floor, Statler-Hilton Hotel)
Electronics-Semiconductors (Abstracts 144-157) 9:00 A.M.-12:00 M.; 1:30-5:00 P.M. (Arabian Ballroom, Hotel Tuller)
Electro-Organic (Abstracts 172-178) 9:00 A.M.-12:00 M.; 1:30-5:00 P.M. (English Room, Ballroom floor, Statler-Hilton Hotel)

Electrothermics and Metallurgy (Abstracts 195-200) 9:00-11:45 A.M. (Italian Room, Hotel Tuller)

Thursday, October 5, 1961

Battery (Abstracts 26-31) 9:00-11:55 A.M. (Wayne Room, Ballroom floor, Statler-Hilton Hotel)

Electrodeposition (Abstracts 107-112) 9:00 A.M.-12:00 M. (Bagley Room, Ballroom floor, Statler-Hilton Hotel)

GENERAL INFORMATION

Convention Headquarters is the Statler-Hilton Hotel, Grand Circus Park at Washington Blvd., Detroit 31, Mich.

Rates per day for rooms with shower are: single, $7.50-12.00; tub and shower $12.50-14.00; double, with shower, $13.00-16.00; with tub and shower $16.50-17.50; twin, with shower $13.50-19.50; with tub and shower, $20.50-29.00; small suites, $25.00 for one, 30.00 for two; large suites for one or two $38.50.

Requests for room reservations should be mailed to the Statler-Hilton Hotel, Grand Circus Park at Washington Blvd., Detroit 31, Mich. If accommodations are not available at the Statler-Hilton, the hotel will make suitable reservations for you at a nearby hotel at a comparable price.

BE SURE to state that you will be attending The Electrochemical Society Meeting. If your wife will be attending, be sure to so indicate on your request for reservations.

The Office of the Secretary is Parlor B, Ballroom floor.
The Detroit Local Section Office is Parlor D, Ballroom floor.
Ladies' Headquarters is the Ivory Room, Ballroom floor.

REGISTRATION

The Registration Desk will be in the Foyer, Ballroom floor Sunday, October 1 through Thursday, October 5, 1961.

The registration schedule is:

Sunday, October 1—2:00-9:00 P.M.
Monday, October 2—7:30 A.M.-5:00 P.M.
Tuesday, October 3—8:00 A.M.-4:00 P.M.
Wednesday, October 4—8:00 A.M.-4:00 P.M.
Thursday, October 5—9:00 A.M.-12:00 M.

Registration fees are:

Technical Session Registration (Men and Women)	
Members	$ 7.00
Nonmember Speakers and Coauthors	7.00
Nonmembers	20.00*
Student Members	Complimentary
Student Nonmembers	2.00
Thursday Only	5.00
Ladies' Registration Fee for Social Events	5.00

* If a nonmember fills out an application form and subsequently is elected to membership in the Society, the difference between the nonmember and member registration fee will be applied to his first year's dues. If his membership begins on January 1 he is entitled to the entire differential; if his membership begins on July 1, he is entitled to half the differential.

Most of the technical sessions will be held in various rooms on the Ballroom floor of the Statler-Hilton Hotel; however

several sessions are scheduled in the Hotel Tuller, Grand Circus Park, across the street from the Statler-Hilton.

INFORMATION ABOUT DETROIT

"The Detroit News" recently circulated a Sunday supplement on the City of Detroit. We have lifted some information therefrom. Detroit's population is about 3,762,000; it is a region with 6438 plants manufacturing everything from gelatin capsules and milk carton machinery to electronic computers, space missiles, and, of course, automobiles. Detroit's assets are considerable: available skilled labor, abundant water supply, and three sources of natural gas; it is one of a few cities whose electric utility is building an atomic reactor to investigate the cheap power hope of the future.

To quote from the News Editorial: "Detroit wasn't really the 'Arsenal of Democracy'; it just turned out more war-winning tools than any other city in the world. It probably isn't the nation's best sports town, though it does have more pleasure boats than any other and (from Ty Cobb to Joe Louis and Gordie Howe) has had its share of champions. While it is true that 55 per cent of its manufacturing employes and 10 per cent of those in non-manufacturing employment belong to unions, it truly doesn't rate the attention it gets when a strike that spreads from Newark, Del., to Los Angeles is reported from the negotiation table in Detroit. Actually, in 1959, all strikes cost Detroit a loss of 55/100ths of 1 per cent of work time."

Detroit is the "birthplace" of the modern shopping center with its Northland Center, the first (built in 1954) and still the largest major regional shopping center in the nation, 110 stores on 161 acres of land, providing parking for 9500 cars. Besides its 27 shopping centers, it boasts an excellent school system, both private and public and has 54 institutions of higher learning in its metropolitan area. There's always something to do in Detroit: Detroit Institute of Art, theaters, baseball, football, basketball, hockey, skiing within an hour's drive of downtown Detroit, "largest fresh-water beach in the world," for example.

SYMPOSIA

The Battery Division has scheduled a Fuel Cell Symposium, Wednesday morning on Fuel Electrodes, Wednesday afternoon on Oxygen Electrodes, and Thursday morning on Fuel Cells, in the Wayne Room, Ballroom floor, Statler-Hilton Hotel. On Tuesday morning and afternoon General Sessions are scheduled in the Tuller Room, Hotel Tuller, 14th floor.

The Battery and the Corrosion Divisions have scheduled a Joint Symposium on Fused Salt Corrosion Monday morning and afternoon in the Tuller Room, Hotel Tuller, 14th floor.

The Corrosion Division has scheduled sessions on Corrosion and Inhibition in Aqueous Solution Tuesday morning; High-Temperature Oxidation Tuesday afternoon; and Surface Structure vs. Corrosion Behavior Wednesday morning and afternoon, all in the Michigan Room, Ballroom floor, Statler-Hilton Hotel.

The Electrodeposition Division has scheduled a Symposium on Effects of Addition Agents in Electrodeposition on Monday, Tuesday, and Wednesday morning, in the Wayne Room, Ball-

room floor, Statler-Hilton Hotel, as well as a Symposium on Electrodeposited Magnetic Films all day Wednesday and Thursday morning, in the Tuller Room, 14th floor, Hotel Tuller, in addition to a few general papers.

Electronics Division-Semiconductor Group has scheduled sessions on: Semiconductor Surfaces Monday morning; Fabrication Technology Monday afternoon; Expitaxial Films all day Tuesday; Intermetallic Semiconductors Wednesday morning; and Elemental Semiconductors Wednesday afternoon; Monday and Tuesday sessions are in the Grand Ballroom, Statler-Hilton Hotel, and the Wednesday sessions are in the Arabian Ballroom, Hotel Tuller.

The Electro-Organic Division has scheduled general sessions on Monday and Wednesday and a Symposium on Organic Electrochemistry in Nonaqueous Solvents on Tuesday; all sessions in the English Room, Ballroom floor, Statler-Hilton Hotel.

The Electrothermics and Metallurgy Division has scheduled a Symposium on Sintering of Refractory Metals and Materials, Tungsten Monday morning, Refractory Metals Monday afternoon, Oxides Tuesday morning, Refractory Compounds Tuesday afternoon, (these in the Bagley Room, Ballroom floor, Statler-Hilton Hotel) as well as a session on Anodic Films and Properties of Carbides Wednesday morning in the Italian Room, Hotel Tuller.

BOARD AND COMMITTEE MEETINGS
(Statler-Hilton Hotel)

Sunday, Oct. 1, 2:00 P.M.—**Board of Directors,** Michigan Room, Ballroom floor.

Monday, Oct. 2, 12:15 P.M.—Luncheon Meeting of the **Membership Committee,** Parlor E, Ballroom floor.

Monday, Oct. 2, 2:00 P.M.—**National Convention Committee,** Parlor E, Ballroom floor.

Monday, Oct. 2, 5:00 P.M.—**Editorial Staff of the Journal,** Parlor F, Ballroom floor.

Monday, Oct. 2, 5:00 P.M.—**Council of Local Sections,** Parlor E, Ballroom floor.

Wednesday, Oct. 4, 12:15 P.M.—Luncheon Meeting of the **General Chairman of Future Meetings,** Parlor E, Ballroom floor.

LUNCHEONS, SOCIAL AFFAIRS, AND BUSINESS MEETINGS OF DIVISIONS

Monday, Oct. 2, 5:30 P.M.—**Electrothermics and Metallurgy Division** Cocktail Hour, Bagley Room, Ballroom floor, Statler-Hilton Hotel.

Tuesday, Oct. 3, 12:15 P.M.—**Battery Division** Luncheon and Business Meeting, Arabian Room, Tuller Hotel.

Tuesday, Oct. 3, 12:15 P.M.—**Electrodeposition Division** Luncheon and Business Meeting, Wayne Room, Ballroom floor, Statler-Hilton Hotel.

Tuesday, Oct. 3, 12:15 P.M.—**Electro-Organic Division** Luncheon and Business Meeting, Parlor F, Ballroom floor, Statler-Hilton Hotel.

Wednesday, Oct. 4, 12:15 P.M.—**Corrosion Division** Luncheon and Business Meeting, Michigan Room, Statler-Hilton Hotel.

GENERAL FUNCTIONS
(Statler-Hilton Hotel)

Symposia Chairmen Breakfasts

Special breakfasts will be held for Symposia Chairmen on Monday through Thursday, October 2 through 5. On the day on which he presides at a technical session, each Symposium Chairman should be in the Cafe Rouge, Lobby floor at 7:45 A.M.

Sunday Evening Social Hour

There will be a Sunday evening social gathering in the Wayne Room, Ballroom floor, from 8 to 10 P.M. All those registered for The Electrochemical Society Meeting are invited to attend.

Monday Evening Mixer

There will be a Monday evening social get-acquainted gathering in the Wayne Room, Ballroom floor, from 8:00 to 10:00 P.M. All those registered for The Electrochemical Society Meeting are invited to attend.

Tuesday Palladium Medal Lecture

On Tuesday, October 3, at 5:00 P.M., Professor H. H. Uhlig will deliver the Palladium Medal Lecture in the Wayne Room, Ballroom floor.

Tuesday Palladium Medal Reception and Banquet

On Tuesday, October 3, a reception and banquet will be held for the Palladium Medalist, Professor H. H. Uhlig. Cocktails will be served from 6:45 to 7:15 in the Ballroom; dinner will be served at 7:15 in the Ballroom.

PLANT TRIPS

Monday afternoon.—Two trips are planned for Monday afternoon, one to Parke, Davis & Co., the other to the Phoenix Project of the University of Michigan, Ann Arbor, a nuclear energy research and development program. Buses will leave the Bagley St. Statler-Hilton Hotel entrance at 1:00 P.M.

Wednesday afternoon.—A trip is planned to the Enrico Fermi Reactor Project of the Detroit Edison Co., Monroe, Mich. No clearance is necessary to visit this plant which generates electricity from nuclear energy. Buses will leave the Bagley St. Statler-Hilton Hotel entrance at 1:00 P.M.

Wednesday evening.—A visit is planned to the International Salt Co.; this trip is for the men only; mining custom does not permit women underground. Buses will leave the Bagley St. Statler-Hilton Hotel entrance at 7:30 P.M.

LADIES' PROGRAM

The ladies are cordially invited to make the Ivory Room, Ballroom floor, Statler-Hilton Hotel, their headquarters during the meeting. A coffee hour will be held from 9:00-10:00 A.M. Monday, Tuesday, and Wednesday.

Sunday, Oct. 1—Registration from 2:00 to 9:00 P.M.

Monday, Oct. 2—Coffee hour from 9:00 to 10:00 in the Ivory Room followed by a trip to Greenfield Village in Dearborn. Buses will leave the Bagley St. Hotel entrance at 10:30 A.M. Lunch will be served at Lovett Hall. If the weather should

be extremely bad, the ladies will visit the Henry Ford Museum instead to see replicas of old shops, genuine antique cars and furniture. Monday evening the ladies may attend the mixer from 8:00-10:00.

Tuesday, Oct 3—Coffee hour from 9:00 to 10:00 A.M. Tuesday morning and afternoon are free to enjoy Detroit's many facilities. Ladies are invited to attend the Palladium Medal Reception and Banquet at 6:45 P.M.

Wednesday, Oct. 4—Coffee hour from 9:00 to 10:00 A.M., followed by a bus tour of Detroit's Cultural Center and downtown. Lunch will be served at J. L. Hudson's during a style show. Buses will leave the Bagley St. Hotel entrance at 10:30 A.M.

COST OF VARIOUS FUNCTIONS

(other than registration)

Please buy tickets as early as possible.

Symposia Chairmen Breakfasts	Standard menu prices
Sunday Evening Social Hour	Complimentary
Monday Electrothermics and Metallurgy Division Cocktail Hour	1.50
Monday Evening Mixer	Complimentary
Division Luncheons	$3.75
Tuesday Palladium Medal Reception and Banquet:	
Men	9.00
Ladies	7.00
Ladies' Social Functions:	
Monday	3.50
Wednesday	1.00
Plant Trips:	
Monday	1.50
Wednesday afternoon	1.75
Wednesday evening	1.25

JOURNALS

Copies of the July, August, and September issues of "The Journal of The Electrochemical Society" will be available at the Registration Desk. The price of a single copy of the "Journal" is $1.70 to members and $2.25 to nonmembers.

EXTENDED ABSTRACTS

The Battery Division is making available an extended abstract booklet containing 1000-word abstracts of most of the papers presented at the Division's Detroit Meeting. Copies of the booklet may be obtained from C. H. Clark, 34 Pleasant Way, Deal, N. J., after Sept. 15 for $2.00 each.

The Electrodeposition Division is preparing an extended abstract booklet containing 1000-word and 2000-word abstracts of the two symposia, Symposium on the Effect of Addition Agents in Electrodeposition and the Symposium on Electrodeposited Magnetic Films. Copies will be available at the Registration Desk or may be obtained by mail after Sept. 1 by sending an order **accompanied by check or money order** for $3.00 to Dan Trivich, Secretary-Treasurer of the Electrodeposition Division, Department of Chemistry, Wayne State University, Detroit 2, Michigan.

DISCUSSION

No recordings will be made of oral discussion. Those contributing to the discussion of a paper and desiring their remarks to be published will be supplied by the symposium chairman with a printed form on which any discussion may be written. These forms should be given to the symposium chairman or to the Managing Editor of the Journal after the session; or they can be mailed to the Managing Editor of the Journal, 1860 Broadway, New York 23, N. Y. The discussion will then be referred to the author for reply. Publication of the discussion and the comments of the author depends on publication of the article in the "Journal."

1962 SPRING MEETING
at the
Statler-Hilton Hotel
LOS ANGELES, CALIFORNIA
May 6, 7, 8, 9, and 10, 1962

Sessions probably will be scheduled on:

Electric Insulation
Electronics-Luminescence and Semiconductors
Electrothermics and Metallurgy
Industrial Electrolytic
Theoretical Electrochemistry

Abstracts for the Los Angeles Meeting *(not exceeding 75 words in length)* must reach Society Headquarters, 1860 Broadway, New York 23, N. Y., *not later than* December 15, 1961 to be included in the Los Angeles program. Please indicate on abstract for which Division's symposium the paper is to be scheduled and underline the name of the author who will present the paper. No paper will be placed on the program unless one of the authors, or a qualified person designated by the authors, has agreed to present it in person. An author who wishes his paper considered for publication in the JOURNAL should send triplicate copies of the manuscript to the Managing Editor of the "JOURNAL," 1860 Broadway, New York 23, N. Y.

Presentation of a paper at a technical meeting of the Society does not guarantee publication in the "JOURNAL." However, all papers so presented become the property of The Electrochemical Society and may not be published elsewhere, either in whole or in part, unless permission for release is requested of and granted by the Editor. Papers already published elsewhere, or submitted for publication elsewhere, are not acceptable for oral presentation except on invitation by a Divisional program Chairman.

Written discussion of a published paper should be submitted within two months following publication of the article in the "Journal." A Discussion Section is published semi-annually in the "Journal."

EMPLOYMENT POSTERS

Companies which desire to recruit employees at the Detroit Meeting will have posters to this effect on a Bulletin Board adjacent to the Registration Desks (see Registration for location). Companies are requested to confine their announcements to this Bulletin Board.

NOTE

Photographing of lantern slides, charts, etc., will be permitted unless specifically prohibited by the speaker. Photo flash and photo floods are prohibited.

MEETING ROOM SCHEDULE

Meeting	Monday		Tuesday		Wednesday		Thursday	
	A.M.	P.M.	A.M.	P.M.	A.M.	P.M.	A.M.	P.M.
Battery			T		B	B	B	
Joint Battery—Corrosion	F	F		F				
Corrosion			D	D	D	D		
Electrodeposition	B	B	B	B	T			
					C	C	C	
Electronics—Semiconductors	A	A	A	A	F	F		
Electro-Organic	E	E	E	E	E	E		
Electrothermics and Metallurgy	C	C	C	C	G			

Statler Hilton Hotel

A—Grand Ballroom
B—Wayne Room
C—Bagley Room
D—Michigan Room
E—English Room

Hotel Tuller

G—Italian Room
F—Arabian Ballroom
T—Tuller Room, 14th floor

Technical Program—Detroit Meeting

Monday A.M., October 2, 1961

BATTERY-CORROSION

Joint Symposium on Fused Salt Corrosion, with Henry Leidheiser presiding

(TULLER ROOM, Hotel Tuller, 14th floor)

9:00 A.M.—Introductory Remarks
9:15 A.M.—"Solutions of Metals in Fused Salts" by Daniel Cubicciotti (Abstract No. 32)
10:00 A.M.—"The Formation Potential of Lithium Chloride" by R. D. Walker, Jr., and W. K. Smith (Abstract No. 33)
10:25 A.M.—"Etching of Silver Single Crystals in Fused Sodium Chloride" by K. H. Stern and Jerome Kruger (Abstract No. 34)
10:50 A.M.—Ten-minute intermission
11:00 A.M.—"Corrosion Associated with the ORNL Fused Salt Fluoride Volatility Process" by A. P. Litman and R. P. Milford (Abstract No. 35)
11:25 A.M.—"Corrosion Behavior of High-Temperature Alloys in Fused Fluoride Mixtures" by J. H. DeVan (Abstract No. 36)
11:50 A.M.—"Corrosion of Metals in Molten Sodium Polyphosphate" by L. F. Grantham, M. A. Hiller, and W. S. Ginell (Abstract No. 37)

ELECTRODEPOSITION

Effects of Addition Agents in Electrodeposition, with D. Trivich presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—Introductory Remarks
9:05 A.M.—"The Role of Addition Agents in Bright Nickel Plating" by H. Brown (Abstract No. 69)
9:50 A.M.—"Kinetics of Electrode Processes in Relation to Electrodeposition" by B. E. Conway (Abstract No. 70)
10:35 A.M.—"Crystal Growth and the Structure of Electrodeposits" by S. C. Barnes, J. P. G. Farr, and H. J. Pick (Abstract No. 71)
11:20 A.M.—"Theories of Addition Agents in Electrodeposition" by Eugenio Bertorelle (Abstract No. 72)

ELECTRONICS-SEMICONDUCTORS

Semiconductor Surfaces, with Willis Adcock presiding

(GRAND BALLROOM, Statler-Hilton Hotel)

9:00 A.M.—"The Study of Some Uncommon Surface Treatments on Germanium" by W. A. Albers, Jr., and A. M. Rickel (Abstract No. 113)
9:20 A.M.—"An Impurity Surface State at the Germanium-Aqueous Electrolyte Interface" by P. J. Boddy and W. H. Brattain (Abstract No. 114)
9:40 A.M.—"Diffusion Masking of Silicon and Germanium by the Evaporation of Silicon Monoxide" by Bernard Krieasman and Karabet Simonyan (Abstract No. 115)

10:00 A.M.—"Open Tube Diffusion of Phosphorus in Silicon" by W. J. Greig and J. C. Sarace (Abstract No. 116)
10:20 A.M.—"Evaluation of the Surface Concentration of Diffused Impurity Layers in Silicon" by O. N. Tufte (Abstract No. 117)
10:40 A.M.—"The Effect of Silicon Surface Treatment on Metallic Impurity Deposition" by G. B. Larrabee (Abstract No. 118)
11:00 A.M.—"Passivated Silicon Devices" by C. H. Li (Abstract No. 119)
11:20 A.M.—"Measurement of the Thickness and Refractive Index of Surface Oxide Films on Semiconductor Materials" by G. R. Booker and C. E. Benjamin (Abstract No. 120)
11:40 A.M.—"Hysteresis in the Large-Signal Field Effect in Semiconductor Surfaces" by D. R. Frankl (Abstract No. 121)

ELECTRO-ORGANIC

General Session, with Christopher Wilson presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Improvement in the Electrolytic Preparation of Iodoform" by R. Ramaswamy, M. S. Venkatachalopathy, and H. V. K. Udupa (Abstract No. 158)
9:30 A.M.—"Electrolytically Regenerated Manganic Sulfate for the Oxidation of Aromatic Hydrocarbons, II. Oxidation of Xylenes to Tolualdehydes" by R. Ramaswamy, M. S. Venkatachalapathy, and H. V. K. Udupa (Abstract No. 159)
10:00 A.M.—"Stereochemistry of Electrolytic Reductions of Butynediol and Cyclohexene-1,2-Dicarboxylic Acid" by George Brownell and C. L. Wilson (Abstract No. 160)
11:00 A.M.—"Electrolytic Formation of Oximes" by Tadao Hayashi and C. L. Wilson (Abstract No. 161)

ELECTROTHERMICS AND METALLURGY

Sintering of Refractory Metals and Materials, I. Tungsten, with W. W. Trainor presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Progress in Sintering," Keynote Address by D. R. Wilder (Abstract No. 179)
9:55 A.M.—"Some Effects of Powder Properties and Consolidation Technique on the Properties of Sintered Tungsten Compacts" by G. C. Bodine (Abstract No. 180)
10:30 A.M.—Twenty-minute intermission
10:50 A.M.—"Activated Sintering of Tungsten" by J. H. Brophy, H. W. Hayden, and J. Wulff (Abstract No. 181)
11:25 A.M.—"Sintering Characteristics of Ultrafine Tungsten Powder" by C. R. McKinsey (Abstract No. 182)

Monday P.M., October 2, 1961

BATTERY—CORROSION

Joint Symposium on Fused Salt Corrosion and High-Temperature Fuel Cells, with E. J. Ritchie presiding

(TULLER ROOM, Hotel Tuller, 14th floor)

2:00 P.M.—"The Thermally Regenerative Liquid Metal Cell" by B. Agruss (Abstract No. 38)

2:25 P.M.—"The Sodium|Tin Liquid-Metal Cell, A Preliminary Study" by R. D. Weaver, S. W. Smith, and N. L. Willmann (Abstract No. 39)

2:50 P.M.—"The Liquid-Metal Cell as an Energy Storage Device for Satellite Application" by E. H. Hietbrink and B. Agruss (Abstract No. 40)

3:15 P.M.—"The Lithium Hydride Electrode" by M. E. Indig and R. N. Snyder (Abstract No. 41)

3:40 P.M.—Ten-minute intermission

3:50 P.M.—"High-Temperature Fuel Cells" by M. L. Kronenberg (Abstract No. 42)

4:15 P.M.—"Electrical and Chemical Characteristics of a Hydrocarbon Fuel Cell of the Fused Carbonate Type" by Y. L. Sandler (Abstract No. 43)

4:40 P.M.—"Ionic Conduction in $(ZrO_2)_{.85}$ $(CaO)_{.15}$" by J. Weissbart and R. Ruka (Abstract No. 44)

ELECTRODEPOSITION

Effects of Addition Agents in Electrodeposition (cont'd), with Seward Beacom presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"Effects of Addition Agents on Physical and Mechanical Properties of Electrodeposits" by H. J. Read (Abstract No. 73)

2:45 P.M.—"X-Ray Determination of Residual Stresses in Electrodeposited Coatings" by G. W. Bush and H. J. Read (Abstract No. 74)

3:15 P.M.—"Electron Microscopic Observations of the Structure of Electroplated Nickel" by R. Weil and H. C. Cook (Abstract No. 75)

3:45 P.M.—"The Influence of Adsorbed Material on the Growth of the Metallic Lattice" by T. P. Hoar (Abstract No. 76)

4:15 P.M.—"Evidence for the Deposition of Very Complex Cations" by F. C. Mathers (Abstract No. 77)

ELECTRONICS-SEMICONDUCTORS

Fabrication Technology, with A. Rose presiding

(GRAND BALLROOM, Statler-Hilton Hotel)

1:30 P.M.—"Electrolytic Slicing of Germanium, II. Characteristics of the {110} and {100} n- and p-type Surfaces" by Sumner Sheff (Abstract No. 122)

1:50 P.M.—"Further Study by Electron Microscopy and Diffraction of Germanium Surfaces from Electrolytic Slicing" by J. J. Oberly (Abstract No. 123)

2:10 P.M.—"Spreading during Capillary Alloying" by Karl Busen, Kurt Lehovec, and Raynor Linzey (Abstract No. 124)

2:30 P.M.—"The Spreading of Molten Indium on Germanium" by A. A. Bergh (Abstract No. 125)

2:50 P.M.—"Characteristics of Porous Vycor as a Moisture Getter in Semiconductor Devices" by M. J. Rand (Abstract No. 126)

3:10 P.M.—"Fabrication of Ultra Fast Switching Tunnel Diodes" by H. C. Schindler (Abstract No. 127)

3:30 P.M.—"Automatic Custom Etching of Tunnel Diodes" by V. Vulcan (Abstract No. 128)

3:50 P.M.—"A Very Low Current Controlled Rectifier; Some Problems of Design and Fabrication" by C. W. Heath W. M. Robinson (Abstract No. 129)
4:10 P.M.—"Photolithography Applied to Silicon Transistor Technology" by J. Spanos (Abstract No. 130)
4:30 P.M.—Recent News Papers. Titles and short abstracts will be available at the meeting.

ELECTRO-ORGANIC

General Session, with Christopher Wilson presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"New Study on the Electrolytic Reduction of Dicyandiamide. An Approach to the Essential Problem on Organic Electrode Process" by Kiichiro Sugino, Keijiro Odo, and Eiichi Ichikawa (Abstract No. 162)
2:30 P.M.—"A Continuous Flow Cell for Electrochemical Synthesis" by M. J. Allen (Abstract No. 163)
3:30 P.M.—"Voltammetric Study of the Kolbe Reaction" by M. J. Allen and W. W. Pierson (Abstract No. 164)
4:30 P.M.—"Anhydrous Voltammetry of the Acetate Ion at a Rotating Platinum Anode" by M. J. Allen and W. W. Pierson (Abstract No. 165)

ELECTROTHERMICS AND METALLURGY

Sintering of Refractory Metals and Materials, II. Refractory Metals, with E. M. Sherwood presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"Fourth Plansee Seminar" by H. A. Johansen (Abstract No. 183)
2:40 P.M.—"Sintering of Pressureless Compacted Tungsten and Molybdenum" by H. H. Hausner and A. R. Poster (Abstract No. 184)
3:20 P.M.—"Sintering of Slip Cast Tungsten" by F. R. Charvat, and G. Reinhardt (Abstract No. 184A)
3:45 P.M.—"Plasma Spraying of Tungsten and Refractory Compounds and the Influence of Sintering on the Physical Properties of These Materials" by Irving Strauss and Robert Steinitz (Abstract No. 185)
4:25 P.M.—"Preparation of F48 Alloy by Means of Hydrostatic Pressing and Vacuum Sintering" by J. W. Pugh and J. F. Cerness (Abstract No. 186)
5:30 P.M.—Electrothermics and Metallurgy Division Cocktail Hour, Bagley Room, Ballroom floor, Statler-Hilton Hotel.

Tuesday A.M., October 3, 1961

BATTERY

General Session with Jeanne Burbank presiding

(TULLER ROOM, Hotel Tuller, 14th floor)

9:00 A.M.—Opening Remarks
9:05 A.M.—"The Operation and Use of Reference Cells" by N. C. Cahoon (Abstract No. 1)

9:25 A.M.—"Studies of Voltaic Cell Discharge by Polarization Oscillography" by R. E. Panzer, W. S. Harris, and W. C. Spindler (Abstract No. 2)
9:50 A.M.—"Determination of the Internal Resistance of Leclanche Cells by Square Wave Method" by Aladar Tvarusko (Abstract No. 3)
10:15 A.M.—"Alkaline Manganese Dioxide-Zinc Batteries" by J. L. S. Daley (Abstract No. 4)
10:40 A.M.—Ten-minute intermission
10:50 A.M.—"Solubility of Silver Oxides in Alkaline Solutions" by Stanley Hills (Abstract No. 5)
11:15 A.M.—"The AgO/Ag_2O Electrode in Alkaline Solutions" by T. P. Dirkse (Abstract No. 6)
11:40 A.M.—"A New Design for the Silver-Cadmium Battery" by P. L. Howard (Abstract No. 7)
12:15 P.M.—Battery Division Luncheon and Business Meeting, Arabian Room, Hotel Tuller.

CORROSION

Corrosion and Inhibition in Aqueous Solution, with R. Powers presiding

(MICHIGAN ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Some Recent Developments in Inhibitor Theory" by Norman Hackerman (Abstract No. 45)
9:25 A.M.—"Corrosion Inhibition by Metallic Cations" by Henry Leidheiser, Jr. (Abstract No. 46)
9:50 A.M.—"Uniform and Pitting Corrosion of 3003 Aluminum in Aerated Citrate Buffer Solution" by E. L. Koehler and S. Evans (Abstract No. 47)
10:15 A.M.—Ten-minute intermission
10:25 A.M.—"The Polarization of Metals in Distilled Water" by F. E. DeBoer, J. E. Draley, and C. A. Youngdahl (Abstract No. 48)
10:50 A.M.—"Effect of Partial Dissolution of the Oxide Film on the Aqueous Corrosion Resistance of Zircaloy-2" by S. Kass and D. B. Scott (Abstract No. 49)
11:15 A.M.—"Corrosion and Electrochemical Behavior of Chromium-Noble Metal Alloys" by N. D. Greene, C. R. Bishop, and M. Stern (Abstract No. 50)

ELECTRODEPOSITION

Effects of Addition Agents in Electrodeposition (cont'd), with D. G. Foulke presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"The Growth of Zinc and Other Electrodeposits" by J. M. Keen and J. P. G. Farr (Abstract No. 79)
9:30 A.M.—"The Effect of Thio-Compounds on the Structure of Copper Electrodeposits" by S. C. Barnes (Abstract No. 80)
10:00 A.M.—"Rotating Disk Electrode Techniques for the Study of Addition Agents" by S. E. Beacom and R. N. Hollyer, Jr. (Abstract No. 81)
10:30 A.M.—"The Effect of Some Addition Agents on the Kinetics of Copper Electrodeposition from a Sulfate Solution, I. Cathode Potential-Current Density Relation" by D. R. Turner and G. R. Johnson (Abstract No. 82)

11:00 A.M.—"The Effect of Some Addition Agents on the Kinetics of Copper Electrodeposition from a Sulfate Solution, II. Rotating Disk Electrode Experiments" by G. R. Johnson and D. R. Turner (Abstract No. 83)
11:30 A.M.—"Some Structural Aspects of Nickel Electroplates" by L. P. Bicelli and G. Serravalle (Abstract No. 84)
12:15 P.M.—Electrodeposition Division Luncheon and Business Meeting, Bagley Room, Ballroom floor, Statler-Hilton Hotel.

ELECTRONICS-SEMICONDUCTORS

Epitaxial Films, with Pei Wang presiding

(GRAND BALLROOM, Statler-Hilton Hotel)

9:00 A.M.—"Epitaxial Silicon Thin Films" by K. J. Miller and R. C. Manz (Abstract No. 131)
9:20 A.M.—"Epitaxial Growth of Silicon" by C. H. Li and D. Scaringella (Abstract No. 132)
9:40 A.M.—"Epitaxial Deposition of Silicon in a Hot-Tube Furnace" by B. E. Deal (Abstract No. 133)
10:00 A.M.—"Properties of Epitaxially Grown Silicon Crystals" by C. J. Marlett and P. A. Newman (Abstract No. 134)
10:20 A.M.—"Impurity Control and Distribution in Epitaxial Silicon Films" by D. Kahng, R. C. Manz, M. M. Atalla, and C. O. Thomas (Abstract No. 135)
10:40 A.M.—"Methods for Measuring Resistivity Gradients and Thicknesses in Epitaxial Films" by J. E. McNamara and H. M. Robertson (Abstract No. 136)
11:00 A.M.—Recent News Papers. Titles and short abstracts will be available at the meeting.

ELECTRO-ORGANIC

Organic Electrochemistry in Nonaqueous Solvents, with S. Wawzonek presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—Introductory Remarks
9:10 A.M.—"Electrode Processes in Acetonitrile" by D. H. Geske (Abstract No. 166)
10:10 A.M.—"The Role of Proton Addition in the Polarographic Reduction of Aromatic Hydrocarbons and Carbonyl Compounds in Dimethylformamide" by P. H. Given (Abstract No. 167)
11:10 A.M.—"Polarography and Voltammetry in Dimethylsulfoxide" by I. M. Kolthoff and T. B. Reddy (Abstract No. 168)
12:15 P.M.—Electro-Organic Division Luncheon and Business Meeting, Parlor F, Ballroom floor, Statler-Hilton Hotel.

ELECTROTHERMICS AND METALLURGY

Sintering of Refractory Metals and Materials, III. Oxides, with Donald Hansen presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Sintering of Oxides" by G. C. Kuczynski (Abstract No. 187)

9:30 A.M.—"A Study of the Sintering Behavior of Some Uranium Dioxide Powders" by J. C. Clayton and L. Berrin (Abstract No. 188)

10:00 A.M.—Twenty-minute intermission

10:20 A.M.—"High-Temperature Electrical Conduction in the System UO_2-ZrO_2" by H. A. Johansen and J. G. Cleary (Abstract No. 189)

10:50 A.M.—"Sintering Kinetics of Linde A Al_2O_3" by C. A. Bruch (Abstract No. 190)

Tuesday P.M., October 3, 1961

BATTERY

General Session, with C. H. Clark presiding

(TULLER ROOM, Hotel Tuller, 14th floor)

2:00 P.M.—"The Runaway Condition in Sealed Nickel-Cadmium Cells and the Characteristics of Sealed Cells with Plastic Electrodes" by A. J. Salkind and J. C. Duddy (Abstract No. 8)

2:25 P.M.—"The 'Vicious Cycle' in Secondary Batteries, A Mathematical Approach" by W. G. Eicke, Jr. (Abstract No. 9)

2:50 P.M.—"Oxygen Evolution from Heavily Doped Nickel Oxide Electrodes" by H. N. Seiger and R. C. Shair (Abstract No. 10)

3:15 P.M.—Five-minute intermission

3:20 P.M.—"Variation of Microstructure in Storage Battery Plates with Method of Forming, I" by A. C. Simon and E. L. Jones (Abstract No. 11)

3:45 P.M.—"Variation of Microstructure in Storage Battery Plates with Method of Operation and Manufacture, II" by A. C. Simon and E. L. Jones (Abstract No. 12)

4:05 P.M.—"Porosity, Air Permeability, and Electrical Resistance of Storage Battery Separators" by W. S. Bundy (Abstract No. 13)

4:25 P.M.—"Battery Design-Current Density Distribution in a Cell" by C. M. Shepherd (Abstract No. 14)

5:00 P.M.—The Palladium Medal Address by H. H. Uhlig in the Wayne Room, Ballroom floor, Statler-Hilton Hotel.

CORROSION

High-Temperature Oxidation, with C. V. King presiding

(MICHIGAN ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"The Role of a Displacement Reaction in the Kinetics of Oxidation of Alloys" by R. L. Levin and J. B. Wagner, Jr. (Abstract No. 51)

2:25 P.M.—"Oxidation Kinetics of $ZrBe_{13}$ and $NbBe_{12}$" by Guy Ervin, Jr. (Abstract No. 52)

2:50 P.M.—"Corrosion of Zirconium Alloys in Superheated Steam" by R. D. Misch and Sherman Greenberg (Abstract No. 53)

3:15 P.M.—Ten-minute intermission

3:25 P.M.—"Oxidation of Refractory Metals as a Function of Pressure, Temperature, and Time: Tungsten in Oxygen" by J. N. Ong, Jr. (Abstract No. 54)

3:50 P.M.—"High-Temperature Oxidation of Fe-Cr Base Alloys, with Particular Reference to Fe-Cr-Y Alloys" by E. J. Felten (Abstract No. 55)
4:15 P.M.—"The Difference Effect and Anodic Behavior of Hafnium Dissolving in Hydrofluoric Acid" by W. J. James, M. E. Straumanis, and J. W. Johnson (Abstract No. 56)
5:00 P.M.—Palladium Medal Address by H. H. Uhlig in the Wayne Room, Ballroom floor, Statler-Hilton Hotel.

ELECTRODEPOSITION

Effects of Addition Agents in Electrodeposition (cont'd), with D. R. Turner presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"The Effect of Addition Agents on the Structure and Physical Properties of Gold Electrodeposits" by D. G. Foulke (Abstract No. 85)
2:30 P.M.—"Radiotracer Studies of the Uptake of Sulfur from Brightener Compounds by Copper and Nickel" by J. J. Hoekstra and Dan Trivich (Abstract No. 86)
3:00 P.M.—"A Study of Laminations in Bright Nickel Electrodeposits" by Nicholas Vanderkooi and Dan Trivich (Abstract No. 87)
3:30 P.M.—"The Effects of Organic Additives on the Kinetics of Tin Deposition" by Ernest Yeager, Frank Hovorka, and Stuart Meibuhr (Abstract No. 88)
4:00 P.M.—"Experimental Observations on the Behavior of Addition Agents in Bright Nickel Plating" by I. R. Bellobono and Eugenio Bertorelle (Abstract No. 89)
4:30 P.M.—"Effect of Addition Agents on the Formation of the Bright Deposit from Zn-, Cd-, and Ag-Cyanide Baths" by Tadao Hayashi and Takeo Ishida (Abstract No. 90)
5:00 P.M.—Palladium Medal Address by H. H. Uhlig, Wayne Room, Ballroom floor, Statler-Hilton Hotel.

ELECTRONICS-SEMICONDUCTORS

Epitaxial Films (cont'd), with C. O. Thomas presiding

(GRAND BALLROOM, Statler-Hilton Hotel)

1:50 P.M.—"Germanium Epitaxial Deposition in a Resistance Furnace" by N. P. Cerniglia and Pei Wang (Abstract No. 136A)
2:10 P.M.—"Surface Effects and Autodoping in Epitaxial Germanium Layers" by E. Matovich and R. J. Andres (Abstract No. 137)
2:30 P.M.—"Evaluation of Germanium Epitaxial Films" by J. R. Biard and S. B. Watelski (Abstract No. 138)
2:50 P.M.—"Some Properties of Epitaxial Germanium" by A. B. Kuper and H. Christensen (Abstract No. 139)
3:10 P.M.—"Vapor Growth of GaAs" by V. J. Lyons and V. J. Silvestri (Abstract No. 140)
3:30 P.M.—"Mechanism for the Vapor Transport of Gallium Arsenide with Hydrogen Chloride" by F. V. Williams and R. A. Ruehrwein (Abstract No. 141)
3:50 P.M.—"An Open Tube Carrier Gas Method for the Epitaxial Growth of Gallium Phosphide" by C. J. Frosch and P. W. Foy (Abstract No. 142)
4:10 P.M.—"Vapor Phase Growth of GaAs and GaP" by R. R. Moest and B. R. Shupp (Abstract No. 143)

4:30 P.M.—Recent News Papers. Titles and short abstracts will be available at the meeting.
5:00 P.M.—Palladium Medal Address by H. H. Uhlig, Wayne Room, Ballroom floor, Statler-Hilton Hotel.

ELECTRO-ORGANIC

Organic Electrochemistry in Nonaqueous Solvents (cont'd), with S. Wawzonek presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"Voltammetry in Liquid Sulfur Dioxide" by P. J. Elving and Elio Vianello (Abstract No. 169)
3:00 P.M.—"The Uses of Electrical Methods in Organometal Systems" by R. E. Dessy, Rudolf Salinger, and Richard Jones (Abstract No. 170)
4:00 P.M.—"Electrochemical Preparation of Polyfluoro Chemicals in Anhydrous HF" by R. D. Dresdner (Abstract No. 171)
5:00 P.M.—Palladium Medal Address by H. H. Uhlig, Wayne Room, Ballroom floor, Statler-Hilton Hotel.

ELECTROTHERMICS AND METALLURGY

Sintering of Refractory Metals and Materials, IV. Refractory Compounds, with A. C. Haskell presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"Sintering of Hafnium Carbide with a Fugitive Metal Binder" by Charles Zalabak (Abstract No. 191)
2:30 P.M.—"Sintering of Pure SiC" by G. R. Watson and J. I. Fredriksson (Abstract No. 192)
3:00 P.M.—Half-hour intermission
3:30 P.M.—"Furnaces, Supports, and Atmospheres for the Sintering of Some Borides, Nitrides, and Silicides" by A. Blum (Abstract No. 193)
4:00 P.M.—"The Sintering of Dense, Fine-Grained Beryllides" by R. S. Truesdale, B. B. Lympany, and W. W. Beaver (Abstract No. 194)
5:00 P.M.—Palladium Medal Address by H. H. Uhlig, Wayne Room, Ballroom floor, Statler-Hilton Hotel.

Wednesday A.M., October 4, 1961

BATTERY

Fuel Cell Symposium: Fuel Electrodes, with Ernest Yeager presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—Introductory Remarks
9:05 A.M.—"Chemisorption in Gas Electrode Reactions" by G. J. Young and J. R. Moser (Abstract No. 15)
9:35 A.M.—"The Electromotive Activity of Electrolytically Deposited Hydrogen at Carbon Electrodes" by H. F. Hunger (Abstract No. 16)
10:00 A.M.—"Chronopotentiometry of the Alcohol Fuel Electrode" by Manuel Shaw and W. J. Subcasky (Abstract No. 17)
10:25 A.M.—Ten-minute intermission

10:35 A.M.—"Voltammetric and Chronopotentiometric Study of the Anodic Oxidation of Methanol, Formaldehyde, and Formic Acid" by R. P. Buck and L. R. Griffith (Abstract No. 18)

11:05 A.M.—"The Electrochemical Oxidation of Methanol in Acid Electrolyte" by D. E. Icenhower and A. P. Bond (Abstract No. 19)

11:30 A.M.—"Raney Type Transition Metals as Fuel Electrode Catalysts" by H. Krupp, H. Rabenhorst, G. Sandstede, G. Walter, and R. McJones (Abstract No. 20)

CORROSION

Surface Structure vs. Corrosion Behavior, with F. W. Fink presiding

(MICHIGAN ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"The Nature of Oxide Films on Single Crystal Iron Whisker Surfaces" by J. V. Laukonis (Abstract No. 57)

9:25 A.M.—"The Isolation of Surface Films from Copper" by A. F. Beck and M. J. Pryor (Abstract No. 58)

9:50 A.M.—"Pitting Corrosion of Stainless Steels" by K. Hino (Abstract No. 59)

10:15 A.M.—Ten-minute intermission

10:25 A.M.—"A Comparative Study of the Morphology of Etched Aluminum Foils" by R. Bakish and E. Z. Borders (Abstract No. 60)

10:50 A.M.—"The Anodic Dissolution of Alloys" by R. F. Steigerwald and N. D. Greene (Abstract No. 61)

12:15 P.M.—Corrosion Division Luncheon and Business Meeting, Michigan Room, Statler-Hilton Hotel.

ELECTRODEPOSITION

Effects of Addition Agents in Electrodeposition (cont'd), with Heinz Gerischer presiding

(TULLER ROOM, Hotel Tuller, 14th floor)

9:00 A.M.—"Mechanism of Addition Agent Reaction in Bright and Leveling Nickel Deposition as Studied with Radioactively Tagged Compounds" by B. J. Riley and S. E. Beacom (Abstract No. 91)

9:30 A.M.—"Effect of Organic Additives on the Hydrogen Absorbed by Iron" by T. C. Franklin and Fujio Matsuda (Abstract No. 92)

10:00 A.M.—"Bismuth Electroplating" by J. G. Beach (Abstract No. 93)

10:30 A.M.—Round-Table Discussion

General Paper

11:30 A.M.—"A Cell for Plating Corrosion Test Panels with Varying Current Densities" by A. A. Johnson (Abstract No. 94)

ELECTRODEPOSITION

Electrodeposited Magnetic Films, with D. C. Wendell, Jr., presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Introduction to Magnetic Phenomena of Soft Magnetic Thin Films" by Rexford Alexander (Abstract No. 95)

9:30 A.M.—"A Method of Preparing Iron-Nickel Films and Some of Their Properties" by P. Kuttner and D. Clemson (Abstract No. 96)

10:00 A.M.—"Stress Reducing Organic Additives in the Sulfate-Chloride Bath for Iron-Nickel Deposition" by I. W. Wolf (Abstract No. 97)

10:30 A.M.—"A Discussion of Electrodep. sited, Cylindrical Film Memory Elements" by T. R. Long (Abstract No. 98)

11:00 A.M.—"Magnetic Properties of a 97Fe-3Ni Thin Film Electroplate" by H. J. White and A. J. Kolk (Abstract No. 99)

11:30 A.M.—"Electrodeposited Magnetic Films for Computer Memories by Jet-Cell Plating in High Magnetic Fields" by J. H. Glaser and W. W. Richardson (Abstract No. 100)

ELECTRONICS-SEMICONDUCTORS

Intermetallic Semiconductors, with P. Pollack presiding

(ARABIAN BALLROOM, Hotel Tuller)

9:00 A.M.—"The Pseudobinary System Mg_2Si-Mg_2Ge" by R. J. LaBotz and D. R. Mason (Abstract No. 144)

9:20 A.M.—"Impurity Distribution in Single Crystal Indium Antimonide" by G. R. Cronin (Abstract No. 145)

9:40 A.M.—"Distribution of Sulfur Impurity in InSb Single Crystals" by M. D. Banus and H. C. Gatos (Abstract No. 146)

10:00 A.M.—"Correlation of Electrical Measurements and Chemical Analyses of Zinc- and Cadmium-Diffused GaAs" by J. F. Black (Abstract No. 147)

10:20 A.M.—"Correlation of Lattice Parameter Measurements and Electrical Measurements in Highly Doped GaAs" by J. F. Black and P. Lublin (Abstract No. 148)

10:40 A.M.—Recent News Papers. Titles and short abstracts will be available at the meeting.

ELECTRO-ORGANIC

General session, with M. J. Allen presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"Polarographic Studies in Acetonitrile and Dimethylformamide, VII. Behavior of Benzyl Halides and Related Compounds" by S. Wawzonek and R. C. Duty (Abstract No. 172)

10:00 A.M.—"Polarographic Reduction of Some Diketones and Pyrones" by R. L. Flurry, R. H. Philp, and R. A. Day, Jr. (Abstract No. 173)

11:00 A.M.—"Electrochemical Reduction of the Benzene Ring" by H. W. Sternberg, Raymond Markby, and Irving Wender (Abstract No. 174)

ELECTROTHERMICS AND METALLURGY

Anodic Films and Properties of Carbides, with J. H. Westbrook presiding

(ITALIAN ROOM, Hotel Tuller)

9:00 A.M.—"Electrochemical Oxidation of Titanium Surfaces" by M. E. Sibert (Abstract No. 195)

9:25 A.M.—"Anodic Formation of Titanium Oxide Dielectric Films" by R. T. Lamoureux (Abstract No. 196)
9:50 A.M.—"A Study of the Anodic Film Formed on Titanium" by M. Tecotzky (Abstract No. 197)
10:15 A.M.—Fifteen-minute intermission
10:30 A.M.—"Silicon Carbide—An Electrolytic Capacitor Material" by B. J. Nicholson, W. Haas, and J. J. Flood (Abstract No. 198)
10:55 A.M.—"Characterization and Properties of UC Crystals Produced by Means of Electron-Beam Zone Melting" by Roger Chang and Harry Nadler (Abstract No. 199)
11:20 A.M.—"Mechanical Properties of Nearly Stoichiometric UC Polycrystals between 1500° and 2000°C" by Roger Chang (Abstract No. 200)

Wednesday P.M., October 4, 1961

BATTERY

Fuel Cell Symposium (cont'd): Oxygen Electrodes, with Donald Tuomi presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—Introductory Remarks
2:10 P.M.—"Oxygen Transport and Reaction Rates at an Air-Depolarized Copper Cathode" by H. C. Weber, H. P. Meissner, and D. A. Sama (Abstract No. 21)
2:40 P.M.—"Characteristics of Oxygen Electrodes" by M. Barak, M. I. Gillibrand, and J. Gray (Abstract No. 22)
3:10 P.M.—"Polarization Behavior of the Oxygen Electrode for Fuel Cells" by P. V. Popat (Abstract No. 23)
3:35 P.M.—Discussion of the above papers
3:45 P.M.—Ten-minute intermission
3:55 P.M.—"The Oxygen Cathode Polarization Characteristics on Porous Carbon and Graphite" by Ernest Yeager, K. V. Rao, A. Kozawa, and Frank Hovorka (Abstract No. 24)
4:30 P.M.—"A Study of the Cathodic Reduction of Oxygen at Carbon Electrodes in Acid Electrolyte" by J. E. Wynn (Abstract No. 25)

CORROSION

Surface Structure vs. Corrosion Behavior (cont'd), with E. Koehler presiding

(MICHIGAN ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"On the Fundamentals of Etching of High-Purity Aluminum Foil for Capacitor Uses" by E. Borders, R. Kornhaas, and R. Bakish (Abstract No. 63)
2:25 P.M.—"Electron Diffraction and Electron Microscopy on Oxide Formed in Single Crystals of Iron at around 200°C" by P. B. Sewell, E. G. Brewer, and M. Cohen (Abstract No. 64)
2:50 P.M.—"The Surface Layer on Passive Iron" by J. L. Ord and J. H. Bartlett (Abstract No. 65)
3:15 P.M.—Ten-minute intermission
3:25 P.M.—"The Effect of Surface Structure on Subsequent Corrosion" by J. H. Greenblatt and J. T. N. Atkinson (Abstract No. 66)

3:50 P.M.—"Structure of Oxide Films Formed on Individual Crystal Grain of Stainless Steels" by Goro Shimaoka (Abstract No. 67)

4:15 P.M.—"Double Layer Capacities of Single Crystals of Gold in Perchloric Acid Solutions" by G. M. Schmid and Norman Hackerman (Abstract No. 68)

ELECTRODEPOSITION

Electrodeposited Magnetic Films (cont'd), with Herman Koretsky presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

2:00 P.M.—"An Introduction to the Meaning of Fundamental Magnetic Terms and Their Significance in Magnetic Recording" by G. Bate (Abstract No. 101)

2:30 P.M.—"Magnetic Properties of Ferromagnetic Metals and Their Alloys Electrodeposited from Sulfamate Solutions" by V. Zentner (Abstract No. 102)

3:00 P.M.—"Magnetic Electrodeposits of Cobalt-Phosphorus" by J. S. Sallo and J. M. Carr (Abstract No. 103)

3:30 P.M.—"The Influence of Internal Stress on the Coercive Force of Thick Films of Cobalt and Nickel Electrodeposits" by R. D. Fisher (Abstract No. 104)

4:00 P.M.—"Further Studies of the Transition Region of a Cobalt Sulfate Electrolyte" by H. E. Quinn (Abstract No. 105)

4:30 P.M.—"An Investigation of the Crystal Structure and Magnetic Properties of Cobalt Nickel Platings from a Combination Sulfate/Chloride Bath with Saccharin as an Additive" by H. F. Quinn, M. Sulich, and J. Manley (Abstract No. 106)

Wednesday Evening Round-Table Discussion. Time and place to be announced.

ELECTRONICS-SEMICONDUCTORS

Elemental Semiconductors, with W. L. Dills presiding

(ARABIAN BALLROOM, Hotel Tuller)

1:30 P.M.—"Nondendritic Ribbon Crystals" by H. Norman, G. Russell, and M. Forney (Abstract No. 149)

1:50 P.M.—"Preparation of High-Purity Silicon from Silane" by C. H. Lewis, M. B. Giusto, H. C. Kelly, and Sidney Johnson (Abstract No. 150)

2:10 P.M.—"The Effect of Heat Treatment, Oxygen, and Structure on the Lifetime of Float Zoned Silicon Crystals" by M. D. Jordan and E. W. Stewart (Abstract No. 151)

2:30 P.M.—"Measurement Errors of Silicon Single Crystals and Slices" by J. S. Miller and V. E. Paulos (Abstract No. 152)

2:50 P.M.—"Resistivity Measurements at Microwave Frequencies" by G. L. Allerton and J. R. Seifert (Abstract No. 153)

3:10 P.M.—"Silicon Crystal Perfection Measurement and Grading" by M. R. Everingham (Abstract No. 154)

3:30 P.M.—"Horizontal Zone Leveling of Silicon" by J. L. Porter and D. M. Lamb (Abstract No. 155)

3:50 P.M.—"Iron-Boron Interactions in Silicon" by W. H. Shepherd and J. Turner (Abstract No. 156)

4:10 P.M.—"Trace-Plating" by W. Rindner and J. M. Lavine (Abstract No. 157)
4:30 P.M.—Recent News Papers. Titles and short abstracts will be available at the meeting.

ELECTRO-ORGANIC

General session, with M. J. Allen presiding

(ENGLISH ROOM, Ballroom floor, Statler-Hilton Hotel)

1:30 P.M.—"Kolbe and Crum Brown-Walker Syntheses with Perfluoroaliphatic Acids" by Sherlock Swann, Jr., and F. H. Baasted (Abstract No. 175)
2:00 P.M.—"Anodic Oxidation in a Heterogeneous System" by H. F. Conway, E. B. Lancaster, V. E. Sohns, and S. Dubic (Abstract No. 176)
3:00 P.M.—"Experimental Study of the Anode Effect in the Electrolysis of Fused Mixtures of Sodium Chloride and Zirconium Tetrafluoride" by C. M. Decroly and Rene Winand (Abstract No. 177)
4:00 P.M.—"Ionic Transference in Pure Molten Sodium Nitrate" by R. J. Labrie and V. A. Lamb (Abstract No. 178)

Thursday A.M., October 5, 1961

BATTERY

Fuel Cell Symposium (cont'd): Fuel Cells, with A. Fleischer presiding

(WAYNE ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—Introductory Remarks
9:05 A.M.—"Internal Resistance in Cation-Membrane Fuel Cells" by E. A. Oster, A. P. Fickett, and P. Chludzinski (Abstract No. 26)
9:30 A.M.—"Transport Processes in Ion-Exchange-Membrane Fuel Cells" by Andrew Dravnieks and J. I. Bregman (Abstract No. 27)
9:55 A.M.—"Hydrogen-Halogen Fuel Cell" by R. M. Lurie and Carl Berger (Abstract No. 28)
10:25 A.M.—"Membrane Properties, Catalyst Type, Graphite Electrode Form—Their Effect on Ion-Membrane Fuel Cells" by R. M. Lurie, Carl Berger, and Ralph Schuman (Abstract No. 29)
10:50 A.M.—Ten-minute intermission
11:00 A.M.—"Radioisotope Activated Fuel Cell Electrodes" by Ricardo Salcedo y Gumucio and Maurice Lang (Abstract No. 30)
11:25 A.M.—"Fuel Cell Batteries with Carbon Electrodes" by E. A. Schumacher and R. J. Bennett (Abstract No. 31)

ELECTRODEPOSITION

Electrodeposited Magnetic Films (cont'd), with Abner Brenner presiding

(BAGLEY ROOM, Ballroom floor, Statler-Hilton Hotel)

9:00 A.M.—"A Method for the Study of Nucleation of Thin Films" by A. E. Cahill (Abstract No. 107)

9:30 A.M.—"Magnetic Properties of Electroless Cobalt Films and Their Application in High Density Digital Recording" by M. A. Foley (Abstract No. 108)

10:00 A.M.—"The Preparation and Magnetic Characteristics of Chemically Deposited Cobalt" by R. D. Fisher and W. H. Chilton (Abstract No. 109)

10:30 A.M.—"Electroless Ni-Co Films with Uniaxial Anisotropy" by J. Bagrowski and M. Lauriente (Abstract No. 110)

General Papers

11:00 A.M.—"The Effect of Pressure on the Electroless Deposition Process" by J. S. Sallo, J. I. Swenson, and J. M. Carr (Abstract No. 111)

11:30 A.M.—"Effect of Binary Alloy Plating on Delayed Brittle Failure of Ultra High Strength Steel" by Walter Beck and E. J. Jankowsky (Abstract No. 112)

Abstracts

BATTERY

Abstract No. 1

The Operation and Use of Reference Cells

N. C. Cahoon, Battery Development Lab., Union Carbide Consumer Products Co., Div. of Union Carbide Corp., P. O. Box 6056, Cleveland 1, Ohio

Reference cells are one of the important tools of the electrochemist, and it is surprising that the literature on the subject is so limited. A review of some of the limitations of reference cells is presented. Suggestions for their effective use in the laboratory are offered. A nomograph permitting the ready conversion of any potential, read against one reference electrode, to the equivalent potential against any one of seven other reference electrodes is described.

Abstract No. 2

Studies of Voltaic Cell Discharge by Polarization Oscillography

R. E. Panzer, W. S. Harris, and **W. C. Spindler,** Chemistry Div., Research Dept., U.S. Naval Ordnance Lab., Corona, Calif.

Investigation of cell polarization was done by direct display of cell potential-time curves on a Tektronix 535 oscilloscope. Utilizing simple circuitry incorporating Western Electric 275B relays, single pulses from voltaic cells were displayed at sweep speeds of 10^{-3} to 10^{-6} sec/cm. From Polaroid photographs delineation of polarization components may be done quite readily. The circuitry employed and examples of the data from cells of various types are shown and discussed.

Abstract No. 3

Determination of the Internal Resistance of Leclanché Cells by Square Wave Method

Aladar Tvarusko, Carl F. Norberg Research Center, Electric Storage Battery Co., Yardley, Pa.

The internal resistance of Leclanché cells was studied on shelf and during discharge by means of a square wave technique. A constant current square wave signal was passed through the test cell and the potential variation across the cell displayed on a high-sensitivity oscilloscope with differential input. The instantaneous voltage drop in the oscilloscope pattern, caused by the leading edge of the constant current square wave, indicated the pure resistance of the Leclanché cells. The internal resistance is independent of the amplitude and frequency of the applied square wave current and of the momentary d-c current. The type of manganese dioxide used in the cathode mix and the composition of the electrolyte influenced the internal resistance of D-size Leclanché cells on shelf and during 4Ω continuous discharge.

Abstract No. 4

Alkaline Manganese Dioxide-Zinc Batteries

J. L. S. Daley, Battery Development Lab., Union Carbide Consumer Products Co., Div. of Union Carbide Corp., P. O. Box 6056, Cleveland 1, Ohio

The MnO_2-Zn-KOH system will, in heavy drain applications, perform at far higher efficiency than its Leclanché counterpart. Low freeze point electrolyte, stable low impedance, and excellent shelf properties, plus a number of novel safety features built into its commercial versions make the alkaline MnO_2-Zn cell potentially very useful.

Abstract No. 5

Solubility of Silver Oxides in Alkaline Solutions

Stanley Hills, Research and Development Dept., Yardney International Corp., 40 Leonard St., New York 13, N. Y.

The solubilities of both monovalent and divalent silver oxides, as determined polarographically over a KOH concentration range of 2-11.7M, vary between 4.5×10^{-4} and 7.5×10^{-4}M. Both oxides display a maximum solubility between 4 and 6M KOH. The solubility, in 45% KOH, of monovalent silver oxide is not affected by the presence of zincate (80 g ZnO/liter). Alkaline silver solutions were analyzed by a simple method, employing a dropping mercury electrode, which is as yet unreported in the literature. Silver, in a metastable valence state greater than one, was found to be soluble in concentrated alkali.

Abstract No. 6

The AgO/Ag_2O Electrode in Alkaline Solutions

T. P. Dirkse, Calvin College, Grand Rapids, Mich.

The standard potential of the $AgO/Ag_2/OH^-$ electrode has been evaluated by direct measurement against a hydrogen electrode. The results appear to be somewhat dependent on the KOH concentration. Comparisons are made with voltages usually associated with the silver-zinc battery.

Abstract No. 7

A New Design for the Silver-Cadmium Battery

P. L. Howard, Yardney Electric Corp., 40 Leonard St., New York 13, N. Y.

For the past number of years the silver-cadmium battery has been designed for use as a normal low-rate charge system. During the past year requirements arose in which batteries would have to be completely charged and recharged with a 1-hr charging time. This has been accomplished. Test programs indicate that between 500 and 1000 cycles can be obtained in a 100-min cycle, of which 35 min is for discharge up to 65% of the cell capacity and the remaining 65 min is used to recharge the battery completely. Tests up to 50% discharge are now between 1500 and 2000 cycles and are still maintaining their total capacity. Other types of batteries are also reported on with the same type characteristics in both the sealed and open types.

Abstract No. 8

The Runaway Condition in Sealed Nickel-Cadmium Cells and the Characteristics of Sealed Cells with Plastic Electrodes

A. J. Salkind and **J. C. Duddy,** Carl F. Norberg Research Center. Electric Storage Battery Co., Yardley, Pa.

The discharge and overcharge performance characteristics of sealed nickel-cadmium cells with plastic (DP) electrodes are presented. These cells operated at high energy density levels on both a weight and volumetric basis. The thermal runaway condition which can occur in sealed nickel cells of all types on constant potential charging is reviewed and discussed.

Abstract No. 9

The "Vicious Cycle" in Secondary Batteries, A Mathematical Approach

W. G. Eicke, Jr., Electrochemistry Section, Electricity Div., National Bureau of Standards, Washington 25, D. C.

Equations relating voltage, temperature, heat capacity, time, and current have been derived for a cell on

constant-potential charge. The equations have been derived assuming the cell to be fully charged and thermally isolated from its surroundings, thereby retaining all of the electrical energy supplied to it. The equations are of the form

$$1/i = A - Bt$$

and

$$1/T = C + D \ln (A - Bt)$$

where i is the current, t the time, T the absolute temperature, and A, B, C, and D are constants depending on the cell size, initial conditions, charging potential, etc. The case where cooling occurs is also considered.

Abstract No. 10

Oxygen Evolution from Heavily Doped Nickel Oxide Electrodes

H. N. Seiger and **R. C. Shair,** Research Lab., Alkaline Battery Div., Gulton Industries, Inc., Metuchen, N. J.

Nickel oxide electrodes were prepared in a nickel matrix. One set of electrodes was doped to 5 at. % with cobalt, and another set with 5 at. % barium. The oxygen evolved after interruption of charge was measured at 1 atm and 100°F. Analysis of data indicates at least three processes involved in the self-discharge mechanism. Doping affects the amounts of oxygen source materials and the evolution rate.

Abstract No. 11

Variation of Microstructure in Storage Battery Plates with Method of Forming, I.

A. C. Simon and **E. L. Jones,** U. S. Naval Research Lab., Washington 25, D. C.

This is a continuation of a previously reported investigation of the microscopy of the forming process. It was found that the microstructure of the plates varied significantly and markedly with changes in temperature, specific gravity, and current density, and that there appeared to be a relationship between the pre-existing sulfate crystal structure and the form of PbO_2 observed after forming.

Abstract No. 12

Variation of Microstructure in Storage Battery Plates with Method of Operation and Manufacture, II

A. C. Simon and **E. L. Jones,** U.S. Naval Research Lab., Washington 25, D. C.

An examination was made of the formed and unformed plates obtained from different manufacturers which revealed that significant differences occurred that could be identified with the source. Characteristics of some of these microstructures appeared to persist

throughout certain types of operation and to relate to performance, although the investigation is too preliminary to substantiate this.

Abstract No. 13

Porosity, Air Permeability, and Electrical Resistance of Storage Battery Separators

W. S. Bundy, Research Labs., National Lead Co., 105 York St., Brooklyn 1, N. Y.

The porosity (void fraction) and pore size distribution of battery separators have been measured by an Aminco-Winslow porosimeter using mercury infusion. Air permeability of these separators has been measured by an Aminco-Frazier permeometer, two "Sheffield porosimeters," and by the Bendsten tester. Resistance measurements in various electrolytes were made with an a-c Kelvin bridge (L&N). By use of Darcy's law and Poisenille's law of fluid flow, the relationship of permeability, electrical resistance, and pore radius is found to be $8K(RA + pt) = r^2pt$, where K, p, R, A, t, and r are Darcy's constant, electrolyte specific resistance, separator electrical resistance, area, thickness, and pore radius, respectively.

Abstract No. 14

Battery Design–Current Density Distribution in a Cell

C. M. Shepherd, U.S. Naval Research Lab., Washington 25, D. C.

An uneven current density distribution in a battery may have a large number of undesirable effects. The current density distribution is a function of electrode height, grid resistance, electrolyte resistance, and polarization. Mathematical solutions have been obtained for those cases where the polarization curves are either linear, logarithmic, cubic, or inverse cubic. The results are presented in graphs which greatly simplify their use in practical battery design.

Abstract No. 15

Chemisorption in Gas Electrode Reactions

G. J. Young and **J. R. Moser,** Alfred University, Alfred, N. Y.

The requirement of rapid chemisorption equilibrium to attain readily reversible gas electrodes necessitates the proper selection of electrode catalysts. In the case of hydrogen, for example, those group VIII metals whose surfaces can be maintained in an active condition give approximately theoretical potentials and can support reaction rates corresponding to current densities of over 10^3 ma/cm^2. However, electrodes catalyzed with the Ib metals do not appear reversible and are strongly activation polarized. Hydrogen chemisorption equilibrium is established rapidly with the group VIII metals,

but not with the group Ib metals. Similar considerations apply to hydrocarbon gas electrodes.

Abstract No. 16

The Electromotive Activity of Electrolytically Deposited Hydrogen at Carbon Electrodes

H. F. Hunger, Primary Battery Branch, Power Sources Div., U.S. Army Signal Research and Development Lab., Fort Monmouth, N. J.

The electromotive activation of hydrogen occurs in an appreciable degree only at electrodes which consist of or contain occluding metals. Further, electromotively active hydrogen can be produced by electrolytical deposition at an occluding or inert electrode. This active hydrogen can be stored in appreciable amounts at inert electrodes, like activated carbon electrodes. The storage capacity depends on the adsorption isotherm of the carbon which is related to the inner surface area.

Abstract No. 17

Chronopotentiometry of the Alcohol Fuel Electrode

Manuel Shaw and W. J. Subcasky, Electrochemical Research Div., Electric Autolite Co., Toledo 1, Ohio

The chronopotentiometric method is adapted to the study of electrochemical oxidation of alcohols. Conditions are described for obtaining well-defined potential-time curves giving reproducible transition times. The method permits the evaluation of transfer coefficients and heterogeneous rate constants, which are used to develop possible mechanisms for the rate-determining step. Interpretation of recovery curves gives additional information on the electrode reactions involved.

Abstract No. 18

Voltammetric and Chronopotentiometric Study of the Anodic Oxidation of Methanol, Formaldehyde, and Formic Acid

R. P. Buck and **L. R. Griffith**, California Research Corp., 200 Bush St., San Francisco 20, Calif.

The anodic oxidation of methanol, formaldehyde, and formic acid on a platinized-platinum electrode was studied by the technique of voltammetry at linearly changing potential at *p*H values from —1 to 16 and over a concentration range of 0.01-0.1M. The *p*H dependence of peak current, overpotential, and kinetic parameters was investigated for each of the three compounds. Mechanisms based on these results, and the supporting chronopotentiometric and product studies, are discussed.

Abstract No. 19

The Electrochemical Oxidation of Methanol in Acid Electrolyte

D. E. Icenhower and **A. P. Bond,** Ordnance Corps, Diamond Ordnance Fuze Labs., Washington 25, D. C.

The electrochemical oxidation of methanol was carried out in acid electrolytes at temperatures from 20° to 75°C using platinized platinum, platinized carbon, and other electrodes. Polarization data were obtained and reaction products were determined at current densities from 1 to 100 ma/cm². Most of the studies were carried out in sulfuric acid, but other electrolytes were investigated.

Abstract No. 20

Raney Type Transition Metals as Fuel Electrode Catalysts

H. Krupp, H. Rabenhorst, G. Sandstede, and **G. Walter,** Battelle Institut, Frankfurt, Germany, and **R. McJones,** Cummins Engine Co., Columbus, Ind.

Porous metal electrodes incorporating Raney catalysts are prepared by powder techniques from transition metals of the first and eighth groups. Half-cell performance is measured in various aqueous electrolytes at temperatures from 25° to 150°C. At 80°C in KOH, hydrogen yields current densities beyond 500 ma/cm², methanol yields well beyond 200 ma/cm², and hexane yields questionable values of a few ma/cm². The methanol reaction approaches complete oxidation.

Abstract No. 21

Oxygen Transport and Reaction Rates at an Air-Depolarized Copper Cathode

H. C. Weber, H. P. Meissner, and **D. A. Sama,** Dept. of Chemical Engineering, Massachusetts Institute of Technology, 77 Massachusetts Ave., Cambridge 39, Mass.

The physical processes by which oxygen is transported to an air-depolarized electrode and the importance of mass transport of oxygen through the electrolyte are discussed. A wiped, rotating, partially submerged cylindrical copper electrode was constructed to eliminate the resistance to oxygen transport posed by the electrolyte. The rate of oxidation of a smooth copper surface leaving a 20% KOH electrolyte was measured with this electrode. The measured oxidation rate is discussed in the light of the rate requirements of the oxygen electrode of a fuel cell.

Abstract No. 22

Characteristics of Oxygen Electrodes

M. Barak, M. I. Gillibrand, and **J. Gray,** Research and Development Labs., Chloride Electrical Storage Co., Ltd., Clifton, Manchester, England

Measurements are recorded of the polarization potentials of some oxygen electrodes at various temper-

atures and current densities. The effects on these characteristics of variations in the composition of the electrolyte are also noted. The reactions were followed by measurements of the consumption of oxygen, and possible reaction mechanisms have been deduced from these results.

Abstract No. 23

Polarization Behavior of the Oxygen Electrode for Fuel Cells

P. V. Popat, Aircraft Accessory Turbine Dept., General Electric Co., Lynn, Mass.

Polarization characteristics of the oxygen electrode in acidic and alkaline media at platinum black and other catalytically active materials for hydrogen-oxygen fuel cells are presented. Experimental evidence strongly suggests that, in the cases studied, the polarization at steady state is associated with the pH changes at each electrode. Open-circuit voltage of the oxygen electrode apparently depends on a number of parameters associated with the electrode, electrolyte, and cell assembly. A tentative explanation for some of the anomalies of the oxygen electrode is advanced.

Abstract No. 24

The Oxygen Cathode Polarization Characteristics on Porous Carbon and Graphite

Ernest Yeager, K. V. Rao, A. Kozawa, and **Frank Hovorka,** Dept. of Chemistry, Western Reserve University, Cleveland, Ohio

The dependence of cathodic polarization on pressure as well as solution parameters has been established in alkaline solutions for various porous carbon and graphite electrodes. With relatively graphitic, low porosity electrodes, the activation polarization associated with the reduction of oxygen has been resolved and the Tafel slope found to be 0.11. Under most circumstances, however, mass transport, peroxide decomposition, and distributed I-R drops within the electrodes are controlling. These results are interpreted in terms of optimizing the performance of porous carbon, oxygen cathodes. [This research sponsored by the Office of Naval Research under Contract Nonr 2391(00)].

Abstract No. 25

A Study of the Cathodic Reduction of Oxygen at Carbon Electrodes in Acid Electrolyte

J. E. Wynn, Primary Battery Branch, Power Sources Div., U.S. Army Signal R and D Lab., Fort Monmouth, N. J.

Theoretical and experimental investigations were made on the oxygen dissolution reaction at porous activated carbon electrodes in acid electrolytes over the pH range 0-7 at 25°C. The current density range for

accurate overpotential measurements was found to be 4.4×10^{-4} to 10^{-3} amp cm^{-2} using a Lugeen-Haber capillary to eliminate IR drop and agitation of the electrolyte to reduce concentration overpotential. At constant current density the mechanism of the complete reduction of molecular oxygen to water on carbon cathodes appeared to be limited by the formation of peroxide intermediates.

Abstract No. 26

Internal Resistance in Cation-Membrane Fuel Cells

E. A. Oster, A. P. Fickett, and **P. Chludzinski,** Aircraft Accessory Turbine Dept., General Electric Co., Lynn, Mass.

The contributions to total internal resistance of each of the cell components are measured and compared with computations for the G.E. cationic-reinforced-membrane fuel cell. On new cells of lower internal resistance, performance increases observed follow predictions.

Abstract No. 27

Transport Processes in Ion-Exchange-Membrane Fuel Cells

Andrew Dravnieks and **J. I. Bregman,** Armour Research Foundation, Technology Center, 10 W. 35th St., Chicago 16, Ill.

Performance of fuel cells utilizing ion-exchange membrane electrolytes depends not only on the polarization at the electrodes and the ionic transport through the membrane, but also on the processes of water metabolism in the cell system. Hence, wetting, electroosmotic, and capillary effects in the membrane and at the electrodes are important. Management of water metabolism is a necessary part in optimization of the membrane cell performance.

Abstract No. 28

Hydrogen-Halogen Fuel Cell

R. M. Lurie and **Carl Berger,** Ionics, Incorporated, Cambridge, Mass.

Cathodic electrolyte composition has been varied over a broad range of acid and halogen concentration. Experimental data, utilizing bromine as an oxidizing agent in this cell, have been obtained and indicate optimal concentration ranges for effective discharge characteristics. Some of the regeneration techniques studied involve electrical charging, heat, and radiation. Results of regenerative performance with respect to catalyst and membrane life are discussed. It appears that regenerable performance of this cell is satisfactory over a period of 1000 hr.

Abstract No. 29

Membrane Properties, Catalyst Type, Graphite Electrode Form—Their Effect on Ion-Membrane Fuel Cells

R. M. Lurie, Carl Berger, and **Ralph Schuman,** Ionics, Incorporated, Cambridge, Mass.

The water content, capacity, and thickness of polystyrene sulfonate membranes have been varied and resistance measurements made and related to ion-membrane fuel cell performance. Polarization data have been obtained for a number of hydrogen and oxygen electrode catalysts and related to potential improvements in catalyst type and electrode configuration in fuel cells. The data have indicated that the major losses in the system are a function of the polarization of the oxygen and hydrogen electrodes while the variation in the physical and chemical form of the membranes is of much less significance in cell performance.

Abstract No. 30

Radioisotope Activated Fuel Cell Electrodes

Ricardo Salcedo y Gumucio and **Maurice Lang,** Yardney Electric Corp., 40 Leonard St., New York 13, N. Y.

Much effort has been spent to develop fuel cell systems both for normal temperature and high-temperature operations. We have viewed the problem from a standpoint of determining the best electrodes and catalysts to reduce polarization to a minimum. To this end we have developed electrodes which incorporate isotopes. Such electrodes show the ability to deliver higher capacity at lower polarization values than the best heretofore prepared electrodes employing conventional catalysts. These electrodes function well at ambient temperatures of 20°-25°C. Experiments with C14 and Pm147 have been conducted using carbon and silver base materials. Concentrations of 10-50 microcuries/cm² have sustained currents up to 200 ma/cm² continuously with polarization values less than 0.2 of a volt. Results of various experiments are discussed.

Abstract No. 31

Fuel Cell Batteries with Carbon Electrodes

E. A. Schumacher and **R. J. Bennett,** Battery Dev. Lab., Union Carbide Consumer Products Co., Div. of Union Carbide Corp., P. O. Box 6056, Cleveland 1, Ohio

Progress in the development of low-temperature, hydrogen oxygen batteries with carbon electrodes is described. The influence of structural parameters and operating conditions on unit cell and multicell battery performance are discussed. Polarization data are given for anodes and cathodes divorced from battery design factors.

BATTERY—CORROSION

Abstract No. 32

Solutions of Metals in Fused Salts

Daniel Cubicciotti, Stanford Research Institute, Menlo Park, Calif.

Certain metals dissolve in their own fused salts. Metals that have been found to show such solubility in their molten halides include the alkali metals, most of the alkaline earth metals, some of the rare earth metals, and certain transition and post-transition metals. Some of the properties of such solutions are reviewed, and their possible bearing on the corrosion of metals by fused salts is mentioned.

Abstract No. 33

The Formation Potential of Lithium Chloride

R. D. Walker, Jr., and **W. K. Smith,** Dept. of Chemical Engineering, University of Florida, Gainesville, Fla.

The standard formation potential of lithium chloride has been measured over the temperature range 615°-794°C in a fused salt galvanic cell using electrodes of lithium electroplated on tungsten and chlorine on graphite and found to be $E^\circ = 3.891\text{-}0.0006714t(^\circ C)$. Stable potentials were not attained for long periods because lithium dissolved in the melt and reacted with the container materials. Derived thermodynamic quantities are: $\Delta G_f^\circ = -89{,}758 + 15.488t$ cal/g mole; $\Delta S^\circ = 15.488$ cal/deg g mole.

Abstract No. 34

Etching of Silver Single Crystals in Fused Sodium Chloride

K. H. Stern and **Jerome Kruger,** National Bureau of Standards, Washington, D. C.

The etching of polycrystalline and single crystal silver surfaces of {100}, {110}, and {111} orientations has been studied in fused sodium chloride at 900°C. Topographies for the different surfaces obtained at 900°C in air and salt were compared. Both smooth electropolished and thermally etched crystals were exposed to the fused sodium chloride. Surface features observed in thermal etching differ from those in fused salt etching; previously thermally etched crystals exposed to fused salt alter their surface structure to that observed when electropolished crystals are exposed to salt alone.

Abstract No. 35

Corrosion Associated with the ORNL Fused Salt Fluoride Volatility Process

A. P. Litman and **R. P. Milford,** Oak Ridge National Lab., Operated by Union Carbide Corp. for the U.S. Atomic Energy Commission, Oak Ridge, Tenn.

Corrosive attack on the two major vessels used in the ORNL fluoride volatility process has often been severe. The process is being studied for recovering uranium from zirconium-uranium nuclear reactor fuel elements. First, the Zr-U is converted to the tetrafluorides in a $NaF\text{-}ZrF_4$ or $NaF\text{-}LiF\text{-}ZrF_4$ melt with HF. INOR-8, Inconel, copper, and nickel have been studied as construction materials for the hydrofluorinator, with INOR-8 preferred. Next, the UF_4 is fluorinated in a second vessel to volatile UF_6, further purified, and collected. 'L'-nickel, INOR-8, HyMu 80, and experimental nickel-rich binary alloys containing Mg, Al, Co, Fe, and Mn have been investigated as fluorinator materials, with 'L'-nickel now in use. Temperatures have ranged from 500°-750°C. Corrosion rate losses during early studies have sometimes exceeded 1 mil/hr as bulk metal losses and/or intergranular attack, pitting, and selective losses of chromium.

Abstract No. 36

Corrosion Behavior of High-Temperature Alloys in Fused Fluoride Mixtures

J. H. DeVan, Oak Ridge National Lab., Oak Ridge, Tenn. Operated for the U.S.A.E.C. by Union Carbide Corp.

The corrosion mechanisms of fused fluoride mixtures are being investigated in conjunction with the development of high-temperature nuclear reactors which employ such salts as fuels and coolants. Methods are presented for predicting corrosion rates in these systems based on the thermodynamic properties of the alloy container and fused salt system. The results of radiotracer studies conducted to demonstrate the proposed corrosion model also are reviewed.

Abstract No. 37

Corrosion of Metals in Molten Sodium Polyphosphate

L. F. Grantham, M. A. Hiller, and **W. S. Ginell,** Atomics International, Div. of North American Aviation, Inc., Canoga Park, Calif.

Corrosion tests of over 60 metals in molten sodium polyphosphate were conducted in connection with the fused oxide reaction program. The rates were determined as functions of alloy composition, metal pretreatment, and phosphate composition. In general, the corrosion rate decreased as the length of the test was increased. The corrosion rates were followed continuously during some tests by observing the intensity of a

volatile corrosion product (P_2) with a mass spectrometer. The results of these examinations are interpreted in terms of diffusion to and through the metal phosphide film formed on the surface of the metal. (This work supported by the U.S.A.E.C.)

Abstract No. 38

The Thermally Regenerative Liquid Metal Cell

B. Agruss, Allison Div., General Motors Corp., Indianapolis 6, Ind.

A unique, thermally regenerative, direct conversion device is described, which is compact, silent, and requires no moving parts. It depends on the reaction of two liquid metals to furnish power and the subsequent thermal decomposition of the reaction product into its original constituents. The theory behind the operation of the system is developed. Laboratory data are given which corroborate the theory for both power production and regeneration. Probable applications are listed.

Abstract No. 39

The Sodium Tin Liquid-Metal Cell, A Preliminary Study

R. D. Weaver, S. W. Smith, and **N. L. Willmann,** Delco-Remy, Div., General Motors Corp., Anderson, Ind.

The experimental results of investigations of cells based on a new concept in energy conversion, the thermally regenerative liquid-metal cell, are presented and compared with predicted performance. Charge-discharge data, electrolyte conductivity data, and life-test data are included. Descriptions of experimental techniques and materials employed are made. Several characteristics of these cells are presented, and recommendations for future work are suggested. It is concluded that these cell systems warrant further intensive study.

Abstract No. 40

The Liquid-Metal Cell as an Energy Storage Device for Satellite Application

E. H. Hietbrink and **B. Agruss,** Allison Div., General Motors Corp., Indianapolis 6, Ind.

Parametric equations were derived which define the minimum weight of an energy storage system operating in a circular orbit about the earth. The system consisted of three components: a liquid-metal cell for generating power during orbit dark time, the required inventory of liquid-metal alloy, and the solar cells necessary to supply power to recharge the cell during orbit light time. The equations define the cell operating voltage which corresponds to minimum system weight in terms of cell operating characteristics, other system component design constants, and orbit light and dark

time. These equations were applied to various liquid metal cell systems to determine the optimum power to weight ratio for each case as a function of orbital altitude.

Abstract No. 41

The Lithium Hydride Electrode

M. E. Indig and **R. N. Snyder,** Delco-Remy, Div. of General Motors Corp., Anderson, Ind.

Results of electrochemical studies on the anodic character of a soluble LiH electrode in a fused LiCl-KCl electrolyte are presented for consideration as a possible high energy, low weight electrode material. Included are constant current polarization studies conducted with different grid materials, coulombic efficiencies based on a proposed two electron oxidation, and the effect of LiH concentration on potential. The experiments are described and possible electrode reactions are suggested with evidence for their justification.

Abstract No. 42

High-Temperature Fuel Cells

M. L. Kronenberg, Research Lab., Union Carbide Consumer Products Co., Div. of Union Carbide Corp., Parma 30, Ohio

Immobilized electrolyte, high-temperature fuel cells of nonconventional design were operated successfully on a wide variety of fuel gases. This design readily permits the incorporation of a working reference electrode and avoids gasketing problems for low-pressure operation. Galvanic utilization of carbon monoxide has been demonstrated whereas galvanic utilization of hydrocarbon fuels appears to depend on cracking or reforming operations.

Abstract No. 43

Electrical and Chemical Characteristics of a Hydrocarbon Fuel Cell of the Fused Carbonate Type

Y. L. Sandler, Research Labs., Westinghouse Electric Corp., Beulah Rd., Churchill Boro., Pittsburgh 35, Pa.

An experimental high-temperature fuel cell is described using alkali carbonates as an electrolyte. The chemical equilibria set up at the fuel electrode and the resulting electrical characteristics are discussed. The cells were operated on natural gas with good output (130 ma/cm^2 at 0.6 v) and at a relatively low temperature (~580°C). The low-temperature reforming reaction with water is used to convert methane almost quantitatively to hydrogen. Causes of instability, unwanted side reactions, and their prevention are discussed.

Abstract No. 44

Ionic Conduction in $(ZrO_2)_{.85}$ $(CaO)_{.15}$

J. Weissbart and **R. Ruka,** Research Labs., Westinghouse Electric Corp., Beulah Rd., Churchill Boro., Pittsburgh 35, Pa.

$(ZrO_2)_{.85}$ $(CaO)_{.15}$ is made the electrolyte of a high-temperature solid electrolyte galvanic cell. Oxygen transported through a cell of the general type Pt, O_2 (P_1) $\|$ $(ZrO_2)_{.85}$ $(CaO)_{.15}$ $\|$ O_2 (P_2), Pt is collected and weighed by a hot zirconium getter suspended from a quartz spring balance. The rate of oxygen transport is compared with current drawn by the cell. This method shows that the ionic conductivity is greater than 98% of the total conductivity at 1000°C.

CORROSION

Abstract No. 45

Some Recent Developments in Inhibitor Theory

Norman Hackerman, Dept. of Chemistry, University of Texas, Austin 12, Texas

The concept of the predominant influence of chemisorption in the inhibition process has been strengthened greatly in the last ten years by an accumulation of evidence which correlates potential shifts, heats of adsorption, and molecular structure with inhibitor efficiency. Specifically, recent experiments have shown a good correlation with pi-orbital character of amino nitrogens in two homologous series of compounds. This appears to strengthen the conclusion of other workers that inhibition by acetylenic compounds is traceable to the pi-orbital character of the electrons forming the triple bond. The important question which must be answered by future research is that of why chemisorption should lead to inhibition. At least two postulates can be made, but experimental proof of either will be quite difficult.

Abstract No. 46

Corrosion Inhibition by Metallic Cations

Henry Leidheiser, Jr., Virginia Institute for Scientific Research, 2820 Grove Ave., Richmond, Va.

Examples of the following types of inhibition by metallic cations are discussed: (*a*) formation of an inert solid solution alloy at the surface; (*b*) formation at the surface of a continuous, elemental coating with high hydrogen overvoltage; (*c*) formation of an inert intermetallic compound; and (*d*) induced formation of an oxide film. The important roles of diffusion in the solid-state and cathodic reactions involving intermetallic compound formation are stressed.

Abstract No. 47

Uniform and Pitting Corrosion of 3003 Aluminum in Aerated Citrate Buffer Solution

E. L. Koehler and **S. Evans** (present address: North American Aviation, Inc., Dept. 591-356, Canoga Park, Calif.), Central Research and Engineering Div., Continental Can Co., Inc., 7622 S. Racine Ave., Chicago 20, Ill.

In aerated citrate buffer solution, corrosion of 3003 aluminum may be either uniform or pitting. Polarization data indicate that uniform attack typically occurs below the limiting diffusion current for oxygen and is controlled by a limiting rate of anodic solution for the film-covered metal; pitting attack typically occurs at the limiting diffusion current for oxygen. With added chloride, pitting attack always occurs and at a rate which is greater than the oxygen limiting diffusion current. Although useful under anaerobic conditions, the polarization resistance method does not appear generally useful for determining corrosion rate under aerated conditions. However, anodic polarization data are useful for determining whether corrosion is of a uniform or pitting nature.

Abstract No. 48

The Polarization of Metals in Distilled Water

F. E. DeBoer, J. E. Draley, and **C. A. Youngdahl,** Argonne National Lab., 9700 S. Cass Ave., Argonne, Ill.

A method of determining the polarization behavior of metals in distilled water has been developed. Difficulties are encountered due to potential gradients in the water, double layer capacity effects, and the influence of the polarizing current on the corroding interface. The procedures used to overcome these difficulties are described, and representative polarization curves are presented.

Abstract No. 49

Effect of Partial Dissolution of the Oxide Film on the Aqueous Corrosion Resistance of Zircaloy-2

S. Kass and **D. B. Scott,** Bettis Atomic Power Lab., Westinghouse Electric Corp., Pittsburgh, Pa.

The aqueous corrosion resistance of corrosion-tested Zircaloy-2 was found to decrease markedly after vacuum heating to dissolve the oxide film partially. Although a complete explanation of the accelerated corrosion is not proposed, several possible mechanisms are presented.

Abstract No. 50

Corrosion and Electrochemical Behavior of Chromium-Noble Metal Alloys

N. D. Greene (present address: Dept. of Metallurgical Engineering, Rensselaer Polytechnic Institute, Troy, N. Y.) **C. R. Bishop,** and **M. Stern** (present

address: Speedway Labs., Linde Co., 1500 Polco St., Indianapolis 24, Ind.) Metals Research Labs., Union Carbide Metals Co., Div. of Union Carbide Corp., Niagara Falls, N. Y.

Alloying chromium with small amounts of platinum, palladium, iridium, rhodium, ruthenium, or osmium markedly improves its corrosion resistance to nonoxidizing acids such as sulfuric or hydrochloric acid. The presence of rhodium, palladium, or osmium has little influence on the resistance of chromium to an oxidizing acid such as nitric acid, while platinum, iridium, or ruthenium greatly increases corrosion rates in this medium. These phenomena can be explained in terms of the electrochemical and corrosion behavior of the metals involved, and are related to the passive and transpassive behavior exhibited by chromium.

Abstract No. 51

The Role of a Displacement Reaction in the Kinetics of Oxidation of Alloys

R. L. Levin and **J. B. Wagner, Jr.**, Hammond Metallurgical Lab., Yale University, New Haven, Conn.

The oxidation of some copper-zinc alloys and a copper-nickel alloy in pure oxygen at 700°C has been studied to determine the occurrence of a displacement reaction of the type.

$$B^{+2} + 2e^- + AO = A^{+2} + 2e^- + BO$$

as proposed by C. Wagner. The formation of BO in the displacement reaction has been shown to occur by observing a change in the oxide scale by metallographic examination and by noting a change in the rate of oxidation as given by the parabolic reaction rate constant. A change in the oxidation rate for several samples was further emphasized by observing the rates during two periods of oxidation which were separated by an isothermal annealing period in an inert gas.

Abstract No. 52

Oxidation Kinetics of $ZrBe_{13}$ and $NbBe_{12}$

Guy Ervin, Jr., Atomics International, Div. of North American Aviation, Inc., P. O. Box 309, Canoga Park, Calif.

A study of the oxidation of $ZrBe_{13}$ and $NbBe_{12}$ in the temperature range 700°-1500°C, using a volumetric technique, has given rates with parabolic time dependence above 800°C, but accelerating rates at 700°C due to fracture and consequent increase of the metal/gas surface area. In the range 1200°-1500°C the oxide coating is normal BeO and the kinetic behavior similar to that for Be metal oxidation, Below 1200°C the oxidation products and kinetics are more complex. (Work supported by U.S.A.E.C.)

Abstract No. 53

Corrosion of Zirconium Alloys in Superheated Steam

R. D. Misch and **Sherman Greenberg,** Argonne National Lab., 9700 S. Cass Ave., Argonne, Ill.

Zirconium alloys now available do not have adequate corrosion resistance for nuclear superheater conditions, such as 540°C and 600 psi steam. Three avenues of approach have been followed in an effort to produce alloys of adequate corrosion resistance: (*a*) compensating effects in the corrosion product; (*b*) promotion of electronic conductance of corrosion product without accompanying deleterious effects; and (*c*) catalysis of the combination of corrosion product hydrogen at the oxide surface. Although it is apparent that the actual situation is more complex than the above simplifications suggest, progress has in fact been made. Alloys have been produced which have corrosion rates of about 1 mil/year. Consideration must be given to the dependence of corrosion rate on heat treatment and to possible embrittlement of alloys by the absorption of corrosion product hydrogen. (Work performed under auspices of U.S.A.E.C.)

Abstract No. 54

Oxidation of Refractory Metals as a Function of Pressure, Temperature, and Time: Tungsten in Oxygen

J. N. Ong, Jr., Research Labs., Aeronutronic, Ford Rd., Newport Beach, Calif.

The rate of attack of tungsten in oxygen (cm/sec) can be represented by:

$$\text{Rate} = \frac{k_1 C_1^{1/2}}{t^{1/2}} + k_2 C_2 + k_3 C_3 + \frac{k_4 C_4^{1/2}}{t^{1/2}} + k_5 C_5$$

where each term in the equation represents the rate due to the following reactions, respectively: (1) $W + O_2 \rightarrow WO_2$ (2) $WO_2 + \frac{1}{2} O_2 \rightarrow WO_3$ (3) $W + \frac{3}{2} O_2 \rightarrow WO_3$ (4) $W + O_2 \rightarrow WO_2$ (5) $WO_2 + \frac{1}{2} O_2 \rightarrow \frac{1}{3} (WO_3)_3$. The k's are rate constants and C's are concentrations of intermediate species which are functions of oxygen pressure. The experimental results of three workers are correlated in the temperature range 500°-1300°C and pressure range 34.0-0.0013 atm oxygen pressure.

Abstract No. 55

High-Temperature Oxidation of Fe-Cr Base Alloys, with Particular Reference to Fe-Cr-Y Alloys

E. J. Felten, Research Lab., General Electric Co., P. O. Box 1088, Schenectady, N. Y.

The oxidation kinetics of iron-chromium alloys containing 25 and 37.5% chromium and small additions of

various Group III and IIIa metals have been measured between 700° and 1200°C. Agreement with the parabolic rate law is observed for all alloys. Iron-chromium binary alloys and those containing small amounts of aluminum or gallium have good oxidation resistance, but poor scaling resistance. Alloys containing yttrium, lanthanum, dysprosium, gadolinum, and erbium exhibit both good oxidation and scaling resistance. Enhanced scaling resistance for alloys containing the rare earth metals is attributed to the formation of internal as well as external oxide.

Abstract No. 56

The Difference Effect and Anodic Behavior of Hafnium Dissolving in Hydrofluoric Acid

W. J. James, M. E. Straumanis, and **J. W. Johnson,** Depts. of Chemical Engineering and Chemistry and of Metallurgical Engineering, School of Mines and Metallurgy, University of Missouri, Rolla, Mo.

Crystal bar hafnium dissolving in HF in the presence of HCl exhibited a positive difference effect, so pronounced as to overbalance completely the hydrogen volume developed by the internal polarization current and to reduce the rate of self-dissolution by an additional 3 $mm^3\ cm^{-2}\ mm^{-1}$ of H_2. The effect was accompanied by a strong shift of the hafnium electrode toward noble values. The results of the difference effect studies together with potential changes on addition of noble metal salts appear to confirm that the dissolution rate is brought about by direct chemical action of molecular HF.

Abstract No. 57

The Nature of Oxide Films on Single Crystal Iron Whisker Surfaces

J. V. Laukonis, Research Labs., General Motors Corp., Warren, Mich.

A topographic study of high-temperature oxidation on iron whiskers has been reported. Currently the oxide films are being removed from the whiskers and studied with an electron microscope. The oxide film replicas of the single crystal surfaces lend themselves to selected area diffraction and to both light and dark field transmission studies. The structure of the single crystal surface and the structure and composition of oxide films formed at temperatures greater than or equal to room temperature are reported.

Abstract No. 58

The Isolation of Surface Films from Copper

A. F. Beck and **M. J. Pryor,** Metallurgical Labs., Olin Mathieson Chemical Corp., 275 Winchester Ave., New Haven 4, Conn.

A method for the isolation of oxide and sulfide films from copper has been developed. This depends on selec-

tive attack at the film-metal interface by a solution containing 3% ammonium acetate and 0.25% bromine in dried methanol. When stripping was performed in contact with dry air, no film dissolution or contamination was detected.

Abstract No. 59

Pitting Corrosion of Stainless Steels

K. Hino, Sumitomo Metal Industries Ltd., Central Research Labs., Amazasaki, Japan

Stainless steels have complicated properties for pitting corrosion. We studied the influencing factors such as heat treatment, working, surface condition, and welding process on pitting corrosion of various stainless steels in dilute solution of hydrochloric acid containing ferric chloride. Moreover, the effect of velocity of corrosion media and the relation between surface structure of metals and pitting corrosion were investigated.

Abstract No. 60

A Comparative Study of the Morphology of Etched Aluminum Foils

R. Bakish, Alloyd Corp., 35 Cambridge Parkway, Cambridge 42, Mass., and **E. Z. Borders,** Republic Foil Corp., Triangle St., Danbury, Conn.

Electron and optical microscopy data on the morphology and capacitance data on commercially available etched high-purity capacitor grade aluminum foils produced in the U.S. and abroad are presented. Attempts are made to establish correlation between morphology and useful capacitance per unit area for high and low voltage application. Related problems are also discussed.

Abstract No. 61

The Anodic Dissolution of Alloys

R. F. Steigerwald and **N. D. Greene,** Corrosion Research Lab., Dept. of Materials Engineering, Rensselaer Polytechnic Institute, Troy, N. Y.

Potentiostatic anodic polarization techniques are used to correlate the dissolution of alloys with their metallurgical structure and the dissolution behavior of their pure components. A broad range of alloys are studied including the systems Sn-Zn, Fe-Cr, Ti-Cr, and Cu-Ti. On the basis of the modern theory of electrode kinetics and modifications of Tammann's rules, general laws are developed for predicting the anodic behavior and corrosion resistance of alloys. The effect of solid-state interaction on dissolution is shown, and the dissolution of intermediate phases and intermetallic compounds is considered.

Abstract No. 62

There is no Abstract No. 62

Abstract No. 63

On the Fundamentals of Etching of High-Purity Aluminum Foil for Capacitor Uses

E. Borders and **R. Kornhaas,** Republic Foil Corp., Triangle St., Danbury, Conn., and **R. Bakish,** Alloyd Corp., 35 Cambridge Parkway, Cambridge 42, Mass.

Studies on the factors underlying morphology of etched foil structures are presented. The importance of coulomb input, current density, and heat treatment of the metal are evaluated for their contribution to morphological effects observed. Experimental results on studies conducted both with single crystals and polycrystals are presented.

Abstract No. 64

Electron Diffraction and Electron Microscopy on Oxide Formed in Single Crystals of Iron at around 200°C

P. B. Sewell, E. G. Brewer, and **M. Cohen,** Div. of Applied Chemistry, National Research Council, Ottawa, Canada

Large grain polycrystalline specimens and single crystals of iron were oxidized in the temperature range of 175°-260°C in 2 cm O_2. Both the amounts of total oxide and Fe_3O_4 formed were very dependent on the orientation of the iron. In general the thickness of Fe_3O_4 was low on surfaces of random orientation and high on surfaces of low index planes. In every case the Fe_3O_4 layer was highly oriented, as shown by electron diffraction and dark field electron microscopy. Particle size of the oxide was of the order of 50Å. At the lower temperature mainly cubic oxide was observed. The formation of α-Fe_2O_3 was dependent on surface preparation, orientation, and temperature.

Abstract No. 65

The Surface Layer on Passive Iron

J. L. Ord and **J. H. Bartlett,** Dept. of Physics, University of Illinois, Urbana, Ill.

The thickness of the layer on passive iron in 2*N* H_2SO_4 increases from about 20Å at the low potential end of the passive region to about 100Å at the upper end. Above the Flade potential the processes leading to activation do not produce an appreciable thinning of the layer. Under a constant current greater than the steady-state value, from which one starts, the rise of potential seems to be associated first with space charge effects and then with layer thickening.

Abstract No. 66

The Effect of Surface Structure on Subsequent Corrosion

J. H. Greenblatt and **J. T. N. Atkinson,** Naval Research Establishment, Dartmouth, Nova Scotia

Specimens of commercially pure aluminum and two special alloys containing iron and nickel have been examined by electron and optical microscope techniques after short periods of exposure to high-temperature pure water. Certain topographical and structural features of the corrosion films have been characterized and studied with particular reference to the presence of second phase particles in the metal. The specialized techniques developed for film examination are described, and the influence of structural features on corrosion behavior is discussed.

Abstract No. 67

Structure of Oxide Films Formed on Individual Crystal Grain of Stainless Steels

Goro Shimaoka, National Research Institute for Metals, 2-300 Nakameguro, Meguro-Ku, Tokyo, Japan

Electron microscope and selected-area diffraction studies were made on the oxide films produced on thin sections (prepared by electropolishing or chemical etching) of 18Cr-8Ni and 25Cr-20Ni stainless steels in the early stages of oxidation at 300°-600°C. The oxide films consisted of spinel-type oxide and some secondary phases. Complete parallel growth of the spinel oxides was observed on the austenitic single crystals.

Abstract No. 68

Double Layer Capacities of Single Crystals of Gold in Perchloric Acid Solutions

G. M. Schmid and **Norman Hackerman,** Dept. of Chemistry, University of Texas, Austin 12, Texas

The differential capacity of the 100 and 110 plane of of a single crystal of gold was measured in 1*N* to 10^{-3}*N*-$HClO_4$, using a single pulse method. No capacity minimum which could be associated with a zero point of charge was found between —1.0 v (~10 ma/cm² cathodic) and +1.4 v *vs.* SCE. The 100 plane shows an adsorption peak at —0.2 v which is absent on the 110 plane. Otherwise no difference in the behavior of the two planes could be detected.

ELECTRODEPOSITION

Abstract No. 69

The Role of Addition Agents in Bright Nickel Plating

H. Brown, The Udylite Research Corp., Warren, Mich.

The structure of various organic compounds used in aqueous electroplating baths is related to their func-

tions in the baths. Principal attention is given to bright nickel plating and the effect on brightness and leveling of sulfonic, carbonyl, and amino groups, multiple bond carbon to carbon and carbon to nitrogen linkages. Possible mechanisms are considered for brightening and leveling based on adsorption and hydrogenation reactions on nickel as catalyst.

Abstract No. 70

Kinetics of Electrode Processes in Relation to Electrodeposition

B. E. Conway, Dept. of Chemistry, University of Ottawa, Ottawa, Ontario, Canada

Following a brief introduction of the basic concepts in electrode kinetics, the elementary processes in electrodeposition are reviewed, including diffusion, adsorption of ions in the double layer, Faradaic ion transfer at the metal interface, surface diffusion of ad-species, electrochemical crystal building steps, and the role of dislocations. Methods for studying ion transfer and diffusion at electrodes are given and the results of kinetic measurements in selected systems (Hg, Cu, Ag) are given. Possible rate-determining steps are discussed as are some fundamental factors determining the role of adsorption of additives at electrodes during electrodeposition.

Abstract No. 71

Crystal Growth and the Structure of Electrodeposits

S. C. Barnes, J. P. G. Farr, and **H. J. Pick,** Joseph Lucas Lab., Dept. of Industrial Metallurgy, The University, Birmingham 15, England

A comparison is made of published observations on the topography of growing electrodeposits and discussion given of the attempts made to correlate the structures produced with the state of the substrate and electrochemical parameters. Attention is drawn to the application of relaxation techniques. The effects of addition agents on crystal growth and the electrochemical reactions at a cathode are considered; the authors will summarize briefly their own experiences and compare these where possible with other published work.

Abstract No. 72

Theories of Addition Agents in Electrodeposition

Eugenio Bertorelle, Preside Instituto Chimico, RHO, Milan, Italy

Various aspects of the theories of addition agents and their influence on the mechanism of electrodeposition are examined fully and discussed. Particular attention is paid to the theory of inhibition, of complex formation and adsorption; the relation with experimentally ob-

servable quantities much as cathode polarization, codeposition of nonmetallic material deriving from addition agents or their decomposition products, is discussed. Some observations on the electrode reactions of addition agents are reported, and the different features concerning some of the industrially important metals are also mentioned.

Abstract No. 73

Effects of Addition Agents on Physical and Mechanical Properties of Electrodeposits

H. J. Read, Dept. of Metallurgy, Pennsylvania State University, University Park, Pa.

The physical properties to be discussed are density, magnetic behavior, and electrical resistivity. Emphasis is placed on the mechanical properties of hardness, tensile strength, ductility, and internal stress, but some attention is given to modulus of elasticity and to fatigue strength. Instances where addition agents are added intentionally to alter properties will be separated, so far as possible, from those cases where the addition agent serves another purpose, such as a brightener, but affects the physical or mechanical properties at the same time.

Abstract No. 74

X-Ray Determination of Residual Stresses in Electrodeposited Coatings

G. W. Bush, Research and Development Dept., National Steel Corp., Weirton, W. Va., and **H. J. Read,** Dept. of Metallurgy, The Pennsylvania State University, University Park, Pa.

Experimental and mathematical procedures were developed and tested for x-ray determination of stress in deposits from data secured with an x-ray diffractometer. The effects of deposit thickness, variations in elastic modulus, and stresses owing to differential thermal expansion between deposit and basis metal have been evaluated. Work on nickel deposits showed stress can be evaluated to ±4,000-6,000 psi and that stress was uniformly distributed over the area irradiated by the x-ray beam.

Abstract No. 75

Electron Microscopic Observations of the Structure of Electroplated Nickel

R. Weil and **H. C. Cook,** Dept. of Metallurgy, Stevens Institute of Technology, Hoboken, N. J.

Electron microscopy using thin films and replicas was employed to study the nature of various structural features due to addition agents, observed in electroplated nickel. The growth mechanisms were investigated by viewing the structure at successive stages in the deposition process. Selected-area diffraction was

used to measure grain sizes and orientations. By observing samples annealed at several temperatures, the locations of foreign substances and their influences on the structure were determined.

Abstract No. 76

The Influence of Adsorbed Material on the Growth of the Metallic Lattice

T. P. Hoar, Dept. of Metallurgy, University of Cambridge, Cambridge, England

Addition agents, whether used for brightening, levelling, refining of grain size, or reduction of tensile stress in electrodeposits, are commonly held to act "by adsorption." The present paper attempts to discriminate between different kinds of adsorption according to the lattice structure and electrochemical characteristics of the metal being deposited, and the size, shape, and polarizability of the molecule being adsorbed. Empirical knowledge of the various kinds of addition agents can thereby be partially codified and explained. Outstanding problems in the field are reviewed briefly.

Abstract No. 77

Evidence for the Deposition of Very Complex Cations

F. C. Mathers, Chemistry Dept., Indiana University, Bloomington, Ind.

This paper gives experimental, but circumstantial, evidence that everything in the bath that has an effect on the nature and composition of the deposited metal must be in the bath in the form of complex cations, the metal itself, the acid ion, addition agents, and other things like "conducting salts," etc. What other explanation is there for the large quantities of tartaric acid and some iron in a silver deposit from the nitrate bath containing ferric nitrate and tartaric acid? The deposition potential of iron is so far from silver that it would seem impossible to deposit any iron otherwise. It is well known that there are many complex cations such as $CdCl^{+}$; therefore, it is logical that all metals and all acids form similar complexes. Ions are now known to be highly solvated so why cannot all the rest of the things in a bath be a part of the cations? How can the acid parts of the various salts get to the cathodes if they are not a part of the cations? Why is the deposit of tin from the fluoride bath always rough and crystalline but smooth from a similar sulfate bath? If bismuth chloride will be dissolved in molten metallic bismuth why not expect cathode deposits to be solutions of the things in the complex cations where the conditions for forming a solution in the metal could be better than when using molten materials?

Abstract No. 78

(There is no Abstract No. 78)

Abstract No. 79

The Growth of Zinc and Other Electrodeposits

J. M. Keen and **J. P. G. Farr,** Joseph Lucas Lab., Dept. of Industrial Metallurgy, The University, Birmingham 15, England

Zinc has been electroplated from acid sulfate solutions onto zinc polycrystalline and single crystalline cathodes, and onto copper single crystal surfaces. The surface features developed are described and are related to those observed on copper electrodeposits in terms of the supposed atomic structure of the substrate surface, of the bulk crystal structure of the deposited metal, and of the growth process. Some experiments on the deposition of lead cadmium and iron are also described and discussed.

Abstract No. 80

The Effect of Thio-Compounds on the Structure of Copper Electrodeposits

S. C. Barnes, Dept. of Industrial Metallurgy, The University, Birmingham, England

Active addition agents in acid copper plating solutions can be divided into two groups: those which increase cathodic overvoltage, and those which lower it. Thio-compounds are in the latter group. Their effects on the characteristics of epitaxial deposits formed on single crystal electrodes have been studied, in conjunction with measurements of cathodic polarization. It is found that pronounced habit changes occur when the addition agents are present in amounts sufficient to affect the overvoltage.

Abstract No. 81

Rotating Disk Electrode Techniques for the Study of Addition Agents

S. E. Beacom and **R. N. Hollyer, Jr.,** Research Labs., General Motors Corp., 12 Mile and Mound Rds., Warren, Mich.

The rotating disk electrode is used as a tool to study the effect of thiourea on the electrodeposition process. Rotating disk theory has been verified for deposition of copper from cupric sulfate solutions in the presence of certain ratios of cupric to inert electrolyte ions. The addition of thiourea produces measurable effects on the several parameters considered. Possible explanations for the role of thiourea are discussed.

Abstract No. 82

The Effect of Some Addition Agents on the Kinetics of Copper Electrodeposition from a Sulfate Solution, I. Cathode Potential-Current Density Relation

D. R. Turner and **G. R. Johnson,** Bell Telephone Labs., Inc., Murray Hill, N. J.

Thiourea, l-cystine, glycine, gelatin, and dextrin were used as addition agents in copper plating from a sulfate

solution. Additions of dextrin up to 0.1 g/l have no effect on the kinetics of copper deposition. All the other addition agents inhibit the rate of copper deposition, and each one has a different behavior. Dextrin enhances the inhibiting effect of thiourea during copper plating. There is evidence that thiourea forms a stable complex with cuprous ions in the plating bath.

Abstract No. 83

The Effect of Some Addition Agents on the Kinetics of Copper Electrodeposition from a Sulfate Solution, II. Rotating Disk Electrode Experiments

G. R. Johnson and **D. R. Turner**, Bell Telephone Labs., Inc., Murray Hill, N. J.

A rotating disk electrode technique has been used to study the effect of thiourea, l-cystine, glycine, gelatin, and dextrin on the kinetics of copper electrodeposition from a sulfate solution. The results indicate that some addition agents function solely by means of an adsorption phenomenon whereas others involve an electrochemical reduction of either the addition agent or a cuprous ion-addition agent complex. The fraction of the total current consumed in reducing the addition agent, or a cuprous ion-addition agent complex, can be determined at any given cathode potential.

Abstract No. 84

Some Structural Aspects of Nickel Electroplates

L. P. Bicelli and **G. Serravalle**, Laboratori di Elettrochimica di Chimica Fisica E di Metallurgia del Politecnico di Milano, Piazza Leonardo da Vinci 32, Milan 132, Italy

Cathodic behavior of nickel polycrystalline and single crystal electrodes, oriented following the (100), (110), and (111) planes, has been investigated in perchlorate and sulfamate baths. The crystallization process of definite thickness deposits has been investigated particularly. X-rays were used to determine the basis and bath influence on the preferred orientations of the deposits, while the kinetic aspects of the electrodeposition processes are studied by means of current efficiency and overvoltage measurements. Some correlations between crystallographic results and overvoltage measurements have been established.

Abstract No. 85

The Effect of Addition Agents on the Structure and Physical Properties of Gold Electrodeposits

D. G. Foulke, Sel-Rex Corp., Nutley, N. J.

Bright gold processes in common use differ from the nonprecious metal baths in that the emphasis has been on the codeposition of metals with the gold, rather than on the use of organic addition agents. There are

potent reasons for this divergent trend as compared to nickel and copper baths as can be shown by photomicrographs before and after heat treatment. Photomicrographs and x-ray diffractograms will indicate the structure of typical bright gold deposits now being plated and they will be related to the deposit analysis.

Abstract No. 86

Radiotracer Studies of the Uptake of Sulfur from Brightener Compounds by Copper and Nickel

J. J. Hoekstra (present address: Dow Chemical Co., Midland, Mich.) and **Dan Trivich**, Dept. of Chemistry, Wayne State University, Detroit 2, Mich.

The uptake of S-35 from thiourea by single crystal spheres of copper and nickel was found to increase with increasing concentration of thiourea in the solution, but showed no preference for any particular crystal face. Allyl sulfonate labelled with S-35 was not taken up by nickel under simple immersion, but was included under condition of nickel electrodeposition. Dissolution of the electrodeposits caused the S-35 to be evolved as H_2S as shown by quantitative analysis and x-ray diffraction. (This work was made possible by a fellowship from the General Motors Research Labs. to J. J. H.)

Abstract No. 87

A Study of Laminations in Bright Nickel Electrodeposits

Nicholas Vanderkooi (present address: General Chemical Div. of Allied Chemical Corp., P. O. Box 405, Morristown, N. J.) and **Dan Trivich**, Dept. of Chemistry, Wayne State University, Detroit 2, Mich.

The laminated structure of bright nickel electrodeposits was studied as a function of current density, pH, cathode shape, and type and concentrations of brighteners, especially the system allyl sulfonate, N-allyl quinaldinium bromide. First-class brighteners (sulfonates) produce laminated electrodeposits; second-class brighteners alone do not, but they do enhance the laminated structure when used with the first-class type. On rough cathodes, equal numbers of laminations are produced on peaks and valleys. The significance of this in levelling is discussed. (This work was made possible by a fellowship from the General Motors Research Labs. to N. V.

Abstract No. 88

The Effects of Organic Additives on the Kinetics of Tin Deposition

Ernest Yeager, Frank Hovorka, and **Stuart Meibuhr** (present address: Applied Research Lab., U.S. Steel Corp., Monroeville, Pa.), Dept. of Chemistry, Western Reserve University, Cleveland 6, Ohio.

The electrode kinetics of tin deposition from simple acid solutions containing various organic substances have been studied by the measurement of the cathodic steady-state polarization as a function of current density. The experimental variables included temperature, concentration of stannous ion, concentration and type of addition agent and type of cathode, pure solid tin and liquid tin amalgam. The results indicate that the rate-determining step is charge transfer.

Abstract No. 89

Experimental Observations on the Behavior of Addition Agents in Bright Nickel Plating

I. R. Bellobono and **Eugenio Bertorelle,** Istituto Chimico, Rho, Milan, Italy

The cathode adsorption of addition agents plays a fundamental part in the studies on the mechanism of bright plating, of levelling, and similar phenomena. Using polarization measurements and the analytical determination of addition agents and their decomposition products at various electrodeposition times, it is possible to obtain important information about the relationship between adsorption and cathode polarization as well as about the influence of the decomposition products which may be present in solution. The experimental researches carried on examine these particular aspects of bright nickel electrodeposition in the presence of addition agents which behave as primary or secondary brighteners. An analytical method which makes use of a spectrophotometric technique for the determination of brighteners in electroplating solutions is also fully described.

Abstract No. 90

Effect of Addition Agents on the Formation of the Bright Deposit from Zn-, Cd-, and Ag-Cyanide Baths

Tadao Hayashi and **Takeo Ishida,** Dept. of Applied Chemistry, University of Osaka Prefecture, Sakai City, Osaka, Japan

Study was made of the effect of addition agents on the formation of bright Zn-, Cd-, and Ag-electrodeposits from cyanide baths. Cathode polarization measurements were carried out in the presence of various addition agents, and the deposits obtained from these baths were analyzed by the electron diffraction method and also by photomicrography in order to elucidate the characteristics of the crystal structure with respect to the formation of the bright deposit.

Abstract No. 91

Mechanism of Addition Agent Reaction in Bright and Leveling Nickel Deposition as Studied with Radioactively Tagged Compounds

B. J. Riley and **S. E. Beacom,** Research Labs., General Motors Corp., Warren, Mich.

Radioactively tagged sodium allyl sulfonate and N-allyl quinaldinium bromide have been used in a con-

tinuing study of the leveling phenomenon associated with the deposition of bright nickel. Further evidence has been obtained to support the previously stated concept of addition agent interaction which accounts for the synergistic effect in leveling brought about by combinations of addition agents.

Abstract No. 92

Effect of Organic Additives on the Hydrogen Absorbed by Iron

T. C. Franklin and **Fujio Matsuda**, Baylor University, Waco, Texas

A study was made of the effect of organic additives on the absorption of electrolytically generated hydrogen by small iron wires and the resultant effect on the embrittlement of the iron. A polarographic technique was used to analyze for the hydrogen, and the embrittlement of iron was studied by a bend test. A linear relationship was observed between the amount of hydrogen absorbed and the embrittlement.

Abstract No. 93

Bismuth Electroplating

J. G. Beach, Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio

Bismuth electrodeposits from bath containing "addition agents" were unsatisfactory toward answering an engineering need for a bismuth nickel-alloy coating. The desired results that were expected from the use of addition agents were obtained, and cogent problems were resolved by the use of periodic reverse-current plating.

Abstract No. 94

A Cell for Plating Corrosion Test Panels with Varying Current Densities

A. A. Johnson, Metal and Thermit Corp., 1700 E. Nine Mile Rd., Detroit 20, Mich.

To enable current density and thickness variations of the magnitude encountered on plated parts to be obtained on the standard flat 4 x 6 in. corrosion test panel, a new laboratory plating cell was designed. The use of the cell for corrosion testing is illustrated, and current density distribution profiles and other cell characteristics are given. The influence of current density and thickness of decorative chromium plate on its crack pattern and on the resulting corrosion protection is discussed.

Abstract No. 95

Introduction to Magnetic Phenomena of Soft Magnetic Thin Films

Rexford Alexander, Burroughs Lab. Research Center, Burroughs Corp., Paoli, Pa.

A simple model of the magnetic properties of ferromagnetic materials is presented for the description of

terms of significance in magnetic thin films work. The anisotropy properties and the dynamics of thin films remagnetization are stressed.

Abstract No. 96

A Method of Preparing Iron-Nickel Films and Some of Their Properties

P. Kuttner and **D. Clemson,** Electronic Instruments Div., Burroughs Corp., Philadelphia, Pa.

Films of 1000Å thickness with excellent anisotropy ($H_K = 1.2$ oe), and coercivity of 0.5 oe have been made reproducibly from an aqueous bath of nickel and iron sulfate with addition agents. Plating is done in a magnetic field. The film composition is 95% Ni, 5% Fe. Without addition agents, the composition and magnetic properties of the films change. However, the magnetic properties cannot be explained on the basis of composition alone.

Abstract No. 97

Stress Reducing Organic Additives in the Sulfate-Chloride Bath for Iron-Nickel Deposition

I. W. Wolf, General Electric Corp., Syracuse, N. Y.

The sulfate-chloride bath has been used for the deposition of magnetic codeposits of nickel and iron. Of special interest have been the very thin films (approximately 1000Å) used for computer memory elements. Stress reduction in these codeposits was achieved by adding saccharin to the solution. However, the role of saccharin was not well understood. In this work, various organic molecules were added to baths in place of saccharin. Few were capable of reducing stress at all detectably and none as well as saccharin. There is strong evidence of a need for a very specific molecular form for stress reduction in this system.

Abstract No. 98

A Discussion of Electrodeposited, Cylindrical Film Memory Elements

T. R. Long, Bell Telephone Labs., Inc., Murray Hill, N. J.

A progress report is presented on a program of electrodepositing Ni-Fe films on a wire substrate for use in a nondestructive, cylindrical film memory. Permalloy films with uniaxial anisotropy are continuously deposited on a 5-mil diameter substrate. Experimental data on the process as well as on the magnetic character of the material are presented. The continuous nature of the process introduces some unique problems of control in the deposition of an alloy whose characteristics are strongly dependent on composition. These problems as well as some compensating advantages are discussed.

Abstract No. 99

Magnetic Properties of a 97Fe-3Ni Thin Film Electroplate

H. J. White and **A. J. Kolk,** Electronics Div., National Cash Register Co., Hawthorne, Calif.

The NCR "Rod" thin film computer memory element employs a 97Fe3Ni alloy deposited on a silvered-glass cane or a BeCu wire of approximately 10 mil diameter. The physical condition of the surface of the silver or the BeCu has been found to be of great importance in determining the magnetic properties of the overplate. The effects of current density, *p*H, plate thickness, and magnetic field applied during plating have been investigated. Unlike permalloy, the magnetic properties were not found to be sensitive to minor variations in alloy composition and were only slightly affected by the level of stress in the plate. The high-speed magnetic switching coefficient of the "Rod" is of a magnitude normally associated with the incoherent rotation mechanism.

Abstract No. 100

Electrodeposited Magnetic Films for Computer Memories by Jet-Cell Plating in High Magnetic Fields

J. H. Glaser and **W. W. Richardson,** Philco Corp., Willow Grove, Pa.

A new method has been developed for plating magnetic films by jet-cell plating technique (Philco propr.) which will allow monitoring of film properties during plating process and terminating operation at optimum condition. The technique allows plating in very high magnetic fields which results in dense films with high output and very good anisotropy. Individual monitored spot plating *vs.* etched film-matrix is evaluated.

Abstract No. 101

An Introduction to the Meaning of Fundamental Magnetic Terms and Their Significance in Magnetic Recording

G. Bate, Product Development Lab., Data Systems Div., International Business Machines Corp., Poughkeepsie, N. Y.

The paper covers terms used in describing the magnetic properties of the two principal types of magnetic memory materials. Those terms include saturation magnetization, remanence, squareness, coercive force, etc., and the physical significance of the parameters in the recording process is discussed.

Abstract No. 102

Magnetic Properties of Ferromagnetic Metals and Their Alloys Electrodeposited from Sulfamate Solutions

V. Zentner, Hughes Aircraft Co., Culver City, Calif.

The magnetic properties of nickel, iron, and cobalt electrodeposited from sulfamate solutions and their

change with conditions of deposition were determined. Alloys of nickel-iron, cobalt-iron, and cobalt-nickel plated from sulfamate systems with systematic variations in bath composition were prepared. The dependence of alloy composition on electrodeposition variables was investigated and the relation between magnetic properties, alloy composition, and crystal structure of the deposits determined.

Abstract No. 103

Magnetic Electrodeposits of Cobalt-Phosphorus

J. S. Sallo and **J. M. Carr,** Honeywell Research Center, Hopkins, Minn.

Electrodeposited films of cobalt-phosphorus have been prepared having coercivities ranging from less than 50 oe to more than 1500 oe. The magnetic properties are independent of the substrate. Chemical composition, crystallographic orientation, and electron microscopy including Bitter Pattern studies are related to the observed coercive force. Conditions for preparing materials within this range of coercivity, including materials with stepped hysteresis loops, are described and related to the chemistry of the system.

Abstract No. 104

The Influence of Internal Stress on the Coercive Force of Thick Films of Cobalt and Nickel Electrodeposits

R. D. Fisher, Physical Research Dept., National Cash Register Co., Dayton 9, Ohio

Nickel and cobalt electrodeposits were prepared at low current density (15 ASF) from simple aqueous solution. The influence of the metal salt ($CoCl_2$ or $NiCl_2$) and saccharin solution concentration on the average stress and microstructure of the cobalt and nickel electrodeposits was investigated in conjunction with measurements of their magnetic properties. The coercive force and stress of the nickel and cobalt electrodeposits were determined and compared at thickness values of approximately 5μ. The results indicate that in certain instances the coercive force of the deposits can be directly correlated with the residual stress.

Abstract No. 105

Further Studies of the Transition Region of a Cobalt Sulfate Electrolyte

H. F. Quinn, Federal Systems Div., International Business Machines Corp., Owego, N. Y.

In conjunction with Croll, the author has previously reported the occurrence of a distinct change in the texture of cobalt electrodeposited from a dilute sulfate bath. With all other parameters held constant, this transition was found to occur in the vicinity of pH 4. On repeating the experiments at smaller pH incre-

ments it has been observed that the texture (and the associated magnetic properties) changes smoothly throughout a transition region from 3.8 to 4.8 pH units. It has been shown that the change in coercivity of the plating is not directly associated with the textural changes. The nature of the alteration in the crystal growth process at the cathode is considered in terms of the theory of Willman and others. In particular, it is shown that the type and surface finish of the substrate are not significant controlling factors in the growth process.

Abstract No. 106

An Investigation of the Crystal Structure and Magnetic Properties of Cobalt Nickel Platings from a Combination Sulfate/Chloride Bath with Saccharin as an Additive

H. F. Quinn, M. Sulich, and **J. Manley,** Federal Systems Div., International Business Machines Corp., Owego, N. Y.

In order to obtain a specific recording characteristic and to provide data for process control we have investigated the crystal texture and magnetic properties of platings prepared in a cobalt chloride/nickel sulfate bath containing saccharin as the additive agent. The composition, texture, and magnetic properties of the platings as a function of varying a-c/d-c ratio for each of 4 pH values are reported.

Abstract No. 107

A Method for the Study of Nucleation of Thin Films

A. E. Cahill, General Electric Corp., Syracuse, N. Y.

Measurements of hydrogen overvoltage are used to determine what fraction of a surface consists of gold and what fraction consists of nickel-iron. When very thin layers of nickel-iron have been electroplated on a gold substrate, it is possible by this means to determine to what extent the nickel-iron is continuous. When the nickel-iron deposits in islands, the hydrogen overvoltage indicates that the solution is in contact with both gold and nickel-iron.

Abstract No. 108

Magnetic Properties of Electroless Cobalt Films and Their Application in High Density Digital Recording

M. A. Foley, Burroughs Lab., Research Center, Burroughs Corp., Paoli, Pa.

Electroless cobalt has been deposited on conductive and nonconductive substrates and the magnetic properties evaluated in respect to thickness, coercive force, and remanence. A coercivity range from 40-500 oe can be obtained by varying the plating conditions. The influence of various substrates on the magnetic properties together with bath pH, agitation, and rate of de-

position has been determined. Crystallographic studies of films of low and high coercivity have been performed. The dynamic evaluation of thin magnetic films (2—100 μin.) in high density recording is discussed.

Abstract No. 109

The Preparation and Magnetic Characteristics of Chemically Deposited Cobalt

R. D. Fisher and **W. H. Chilton,** Physical Research Dept., National Cash Register Co., Dayton 9, Ohio

Chemically reduced cobalt films on Mylar have been prepared and their magnetic properties evaluated with respect to squareness, coercive force, remanent and saturation magnetization. The influence of deposition parameters and thickness on the microstructure (grain size and crystal orientation) of the cobalt films was investigated in conjunction with measurement of their magnetic properties. The coercive force may be varied from 200 to 600 oe depending on experimental conditions. The magnitude of the coercive force for a specific thickness is dependent on the grain development and grain size of the film. The squareness (Br/Bm) is dependent on the thickness and crystal orientation of the film. The average saturation flux density is 10,000 gauss.

Abstract No. 110

Electroless Ni-Co Films with Uniaxial Anisotropy

J. Bagrowski and **M. Lauriente,** Air Arm Div., Westinghouse Electric Corp., Baltimore 3, Md.

This study covers an extension of the work by Heritage and Walker on Ni-Co films produced by chemical reduction with uniaxial anisotropy. Of significance was the anomalously low coercive force disclosed for the Ni-Co films. The extension of this work involves an attempt to understand the basic mechanism for the anisotropy.

Abstract No. 111

The Effect of Pressure on the Electroless Deposition Process

J. S. Sallo, J. I. Swenson, and **J. M. Carr,** Honeywell Research Center, Hopkins, Minn.

Hydrostatic pressure is shown to alter the rate of electroless nickel deposition. Two classes of acid baths exist, depending on whether the rate is increased or decreased. This difference explains conflicting literature reports. One of these systems is shown to be diffusion controlled. In the other case, the results indicate that the rate is dependent on the active catalytic area which is exposed to the system and free of hydrogen gas.

Abstract No. 112

Effect of Binary Alloy Plating on Delayed Brittle Failure of Ultra High Strength Steel

Walter Beck and **E. J. Jankowsky**, Aeronautical Materials Lab., Naval Air Materials Lab., Philadelphia 12, Pa.

In a number of applications, involving high strength steel, tin-cadmium alloy plating has been substituted for cadmium plating. In this study, notched specimens of high strength steel were plated with tin-cadmium alloys, produced by codeposition of the components from a fluoborate bath, or by thermal interdiffusion of two separately plated metal layers from a stannate and a cyanide bath. The marked differences found in the susceptibility to delayed cracking were ascribed to differences in the hydrogen content of specimens plated from baths with different compositions, and discussed in the light of the kinetics of crack propagation in delayed failure, put forward by Troiano.

ELECTRONICS—SEMICONDUCTORS

Abstract No. 113

The Study of Some Uncommon Surface Treatments on Germanium

W. A. Albers, Jr., and **A. M. Rickel**, Research Labs., Bendix Corp., Southfield, Mich.

A number of uncommon surface treatments have been investigated on homogeneous germanium single crystals. Simultaneous measurements of a-c field effect and surface recombination velocity as functions of surface potential have been performed in order to evaluate the electrical nature of the resulting surfaces. The effect of surface preparation prior to treatment and crystallographic orientation effects have been included in the investigations. The characterization of a number of surface treatments which show potentialities as possible stabilization and/or passivation treatments is reported.

Abstract No. 114

An Impurity Surface State at the Germanium-Aqueous Electrolyte Interface

P. J. Boddy and **W. H. Brattain**, Bell Telephone Labs., Murray Hill, N. J.

Measurements of the capacity of the germanium-aqueous electrolyte interface in the region of *p*H 7 show negligible additional capacity due to surface states for properly cleaned solutions. Surface recombination velocity (s) is small (or zero) over a range of surface potential (ψ_s). Traces of Cu^{++} (ca. 10^{-5}M) added to the solution increase the capacity and s over certain ranges of ψ_s. These data indicate a recombination center about 50 mv below the center of the gap when Cu^{++} is present.

Abstract No. 115

Diffusion Masking of Silicon and Germanium by the Evaporation of Silicon Monoxide

Bernard Kriegsman and **Karabet Simonyan,** General Instrument Corp., Hicksville, N. Y.

A technique has been developed to mask the diffusion of impurities into silicon and germanium. This technique involves the vacuum evaporation of silicon monoxide onto the particular substrate. In silicon it offers the advantage of forming a continuous and a smooth diffusion mask at low temperatures. This avoids the necessity of thermally growing the mask which can cause an impurity redistribution at the silicon-silicon dioxide interface. In germanium it enables one to make double diffused and planar transistors.

Abstract No. 116

Open Tube Diffusion of Phosphorus in Silicon

W. J. Greig and **J. C. Sarace,** Semiconductor & Materials Div., Radio Corp. of America, Somerville, N. J.

Experimental data are presented for phosphorus diffusion in silicon employing an open tube carrier gas system. Using P_2O_5 as the diffusant source, the surface concentration is calculated in terms of the system parameters, namely, effects of source age, source temperature, silicon temperature, carrier gas, and carrier gas flow rate. Surface concentrations are calculated from sheet resistance and junction penetration measurements using the curves of Backenstoss. Data obtained from an evaluation of the diffusion coefficient calculated assuming a complementary error function distribution are given showing a dependence on the surface concentration and wafer surface condition. Anomalous results obtained from effects of source temperature on surface concentration and the diffusion coefficient as a function of surface concentration also are discussed.

Abstract No. 117

Evaluation of the Surface Concentration of Diffused Impurity Layers in Silicon

O. N. Tufte, Honeywell Research Center, Hopkins, Minn.

The relation between the surface concentration of a diffused layer and the electrical conductivity and Hall coefficient of the layer has been calculated for a complementary error function impurity distribution in silicon. The results show that the Hall coefficient is very nearly independent of the mobility values used in the calculation. This allows accurate values of the surface concentration to be determined without knowing exactly the variation of the mobility with impurity concentration. An experimental comparison of the surface concentration values obtained by the conductivity and Hall effect methods is made.

Abstract No. 118

The Effect of Silicon Surface Treatment on Metallic Impurity Deposition

G. B. Larrabee, Central Research Labs., Texas Instruments Inc., Dallas, Texas

Electrochemical studies of various silicon surfaces show differences in electrochemical potential which can be explained by the mode of formation of the oxide layer on the surface. Radiochemical studies substantiate the electrochemical findings and show why the condition of the silicon surface has such a pronounced effect on the extent of impurity contamination. Autoradiographic studies of the manner in which the metallic impurities are distributed over the various surfaces give additional evidence to explain this phenomenon.

Abstract No. 119

Passivated Silicon Devices

C. H. Li, Semiconductor Div., General Instrument Corp., 600 W. John St., Hicksville, N. Y.

Passivated surface is considered as a hermetic, large-area silicon-to-oxide joint or seal. The mechanical, electrical, and chemical requirements of this seal are discussed, with emphasis on oxide thickness, structure, and composition. Experimental data are given showing why and when on thermal cycling the oxide cracks and silicon deteriorates. A low-temperature passivating method has been developed giving units completely inert to ambient gases, organic solvents, or even various metallic and nonmetallic ions.

Abstract No. 120

Measurement of the Thickness and Refractive Index of Surface Oxide Films on Semiconductor Materials

G. R. Booker and **C. E. Benjamin,** Research Labs., Westinghouse Electric Corp., Beulah Rd., Churchill Boro., Pittsburgh 35, Pa.

The application of monochromatic-light interferometry to the measurement of the thickness and refractive index of surface oxide films on semiconductor materials is described. Interference fringe patterns may be obtained by comparing either the top surface or the bottom surface of a wedge-shaped step in the oxide film with an external reference plane, or by comparing the two surfaces of the wedge with one another. Capabilities and accuracies obtainable using these three systems are discussed. A novel method is described for the rapid measurement of oxide film thicknesses on silicon; the method uses simple equipment and is independent of surface roughness.

Abstract No. 121

Hysteresis in the Large-Signal Field Effect in Semiconductor Surfaces

D. R. Frankl, General Telephone and Electronics Labs., Inc., Bayside 60, N. Y.

The finite generation and recombination rates of excess minority carriers can lead to large hysteresis effects even at frequencies $<<(2\pi\tau)^{-1}$ in large-signal field effect measurements. The hysteresis loop occurs on the inversion-layer side of the conductance *vs.* field curve and widens with increasing frequency and with increasing minority carrier lifetime in the sample. The importance of this effect is that it may lead to appreciable errors in certain point-by-point measuring techniques. Experimental results on germanium and a simple theoretical treatment are presented.

Abstract No. 122

Electrolytic Slicing of Germanium, II. Characteristics of the {110} and {100} n- and p-type Surfaces

Sumner Sheff, Semiconductor Div., Raytheon Co., Newton, Mass.

A metallographic study was made of both sides of {110} and {100} n- and p-type germanium wafers produced by the electrolytic slicing technique. Etch pits were observed on the n-type surfaces which are closely related to those found on the {111} surfaces. These pits extend far into the germanium wafers in one of the <100> directions depending on the side being examined. An explanation of this observation is proposed. The effects of illumination, slice thickness, current density, electrolyte flow rate, and concentration are also discussed. Electron diffraction, electron micrographic, and field effect data for these surfaces are presented in the two following papers.

Abstract No. 123

Further Study by Electron Microscopy and Diffraction of Germanium Surfaces from Electrolytic Slicing

J. J. Oberly, Research Div., Raytheon Co., Waltham, Mass.

The study of germanium surfaces produced by electrolytic slicing has been extended to (100) and (110) surfaces. There is no indication of surface damage, and the surfaces of p-type Ge or of n-type Ge sliced under intense illumination are generally free from pits. The deep pits or "pipes" in unilluminated n-type Ge which have been attributed to avalanche breakdown are partially filled with particles identified as Ge. The randomly oriented Ge film previously observed on n-type (111) surfaces probably consisted of these particles.

Abstract No. 124

Spreading during Capillary Alloying

Karl Busen, Kurt Lehovec, and **Raynor Linzey,** Sprague Electric Co., North Adams, Mass.

This paper describes experiments carried out on (111) oriented germanium which were in contact with liquid indium. It was found that, in spite of a lateral restriction of the indium by the walls of a quartz capillary, there was spreading over the germanium surface, depending on the previous mechanical surface treatment. However, the maximum spreading is limited by the diffusion rate of dissolved germanium in radial direction toward the capillary.

Abstract No. 125

The Spreading of Molten Indium on Germanium

A. A. Bergh, Bell Telephone Labs., Inc., Allentown, Pa.

Factors influencing the spreading of molten indium on germanium oriented parallel to the <111> plane have been investigated. The surface tension of indium as a function of temperature and germanium content has been determined and the effect of the gaseous ambient and surface conditions of germanium studied. It was found that the latter has a more pronounced influence on spreading than the change in the surface tension of indium.

Abstract No. 126

Characteristics of Porous Vycor as a Moisture Getter in Semiconductor Devices

M. J. Rand, Bell Telephone Labs., Inc., Allentown, Pa.

The moisture adsorption capacities of porous Vycor are presented as adsorption isotherms at 30°-150°C with coverages up to 0.3 monolayers. The thermodynamics, kinetics, and mechanism of the adsorption are considered. Vacuum bakeout and hot gas flushing are compared as activation procedures. Equations are developed which predict the changes in an enclosed Vycor-containing ambient as a function of temperature.

Abstract No. 127

Fabrication of Ultra Fast Switching Tunnel Diodes

H. C. Schindler, General Instrument Corp., 600 W. John St., Hicksville, N. Y.

Tunnel diodes are finding increasing application as fast switching devices in control systems and computers. By optimizing the doping level and junction area, tunnel diodes have been prepared with unusually high peak current to capacity ratios which switch in as little as 0.5 manoseconds. 1 ma units of this type in a standard T018 package oscillate at frequencies up to 1.5 kmc. This paper presents a discussion of the

manufacturing techniques employed to obtain these results as well as a correlation between doping level, junction area, and switching speed.

Abstract No. 128

Automatic Custom Etching of Tunnel Diodes

V. Vulcan, General Instrument Corp., 600 W. John St., Hicksville, N. Y.

An automatic electrolytic etching process has been invented for tunnel diodes capable of producing units with any peak current between 100 ma and 250 ma with a tolerance of ±2%. Any starting material may be used. Both analog and digital techniques have been employed in the design of the apparatus. Novel detector and feed-back circuitry forces the pulsed etching current to slave to the instantaneous sampled peak current, thus preventing overetching or burn-out. A tunnel diode gate automatically rejects high contact resistance units. High peak-to-valley ratios and low series resistances are preserved, resulting in desirable etch contours.

Abstract No. 129

A Very Low Current Controlled Rectifier; Some Problems of Design and Fabrication

C. W. Heath and **W. M. Robinson,** Electronics Components Div., General Electric Co., Auburn, N. Y.

The ZJ-203 very low current silicon controlled rectifier is housed in the standard JEDEC TO-5 transistor package. The device employs a welded case, glass-to-metal seals, hard solders for pellet mounting, and a painted external surface for maximum heat dissipation. Design objectives are discussed which have resulted in a PNPN device with very high gate sensitivity, blocking voltages up to 400 v, and extremely uniform characteristics. Some problems and unique features of design and fabrication are discussed. Among these are a relatively new ultrasonic method of bonding ohmic contacts to the silicon pellet, and the investigation of a forward voltage drop problem and its solution through a diffusion operation. Slides will be shown depicting devices construction and electrical characteristics.

Abstract No. 130

Photolithography Applied to Silicon Transistor Technology

J. Spanos, Hoffman Semiconductor Division, El Monte, Calif.

Photolithography as it applies to silicon technology is defined. The relationship of photolithography to device construction is specifically shown. Various techniques of mask making are compared as an auxiliary function. Specific problems in the use of photopolymeric materials are discussed from both a production and laboratory point of view. Device applications are shown.

Abstract No. 131

Epitaxial Silicon Thin Films

K. J. Miller and **R. C. Manz,** Bell Telephone Labs., Inc., Murray Hill, N. J.

The growth of epitaxial silicon films of controlled type, resistivity, and thicknesses for semiconductor devices is described. The surface texture, film crystal structure, and electrical properties are discussed and related to growth conditions.

Abstract No. 132

Epitaxial Silicon Thin Films

C. H. Li and **D. Scaringella,** Semiconductor Div., General Instrument Corp., Hicksville, N. Y.

An improved and reproducible method for epitaxial growth of silicon is described. The equipment is compact, simple, reproducible, and has an improved gas-mixing system. The process allows shorter time for preparing the first run and making each subsequent run. The layers produced are planar and uniform from center to edge. Under 500X magnification, they show no pits, oxides, pyramids, growth scales, or crystal structures. A description is given of the characteristics of diodes and transistors fabricated from this material.

Abstract No. 133

Epitaxial Deposition of Silicon in a Hot-Tube Furnace

B. E. Deal, Research and Development Dept., Rheem Semiconductor Corp., Mountain View, Calif.

A method has been developed for epitaxially depositing single crystal silicon in a conventional hot-tube furnace. Silicon is deposited on a silicon substrate by the reduction of a silicon halide at temperatures in excess of 1200°C. A modification of the gas inlet system prevents decomposition of the silicon halide before contacting the silicon substrate. Several silicon slices can be treated simultaneously with good reproducibility of deposit thickness and resistivities.

Abstract No. 134

Properties of Epitaxially Grown Silicon Crystals

C. J. Marlett and **P. A. Newman,** United States Army Signal Research and Development Lab., Fort Monmouth, N. J.

Resistivity, Hall mobility, lifetime, and carrier concentration have been measured as a function of temperature from 80° to 500°K on a number of epitaxially grown n- and p-type silicon crystals having carrier concentrations between 10^{15} and 10^{20}/cm^3. Similar measurements have also been made on two layer structures of alternate p- and n-type material. The dislocation density of the samples was also observed.

Abstract No. 135

Impurity Control and Distribution in Epitaxial Silicon Films

D. Kahng, R. C. Manz, M. M. Atalla, and **C. O. Thomas,** Bell Telephone Labs., Inc., Murray Hill, N. J.

A study of the epitaxial growth of n-type silicon on n+-type substrates using a doped pedestal as a means of resistivity control in the film is described. Data are presented on the resistivity profiles obtained with the films and their relation to various parameters. Results are also presented on the diffusion of various impurities in epitaxial films and compared with diffusion in bulk silicon. The technique for obtaining the details of the doping profiles within the films also is described.

Abstract No. 136

Methods for Measuring Resistivity Gradients and Thicknesses in Epitaxial Films

J. E. McNamara and **H. M. Robertson,** Materials Research Dept., Semiconductor Products Div., Motorola, Phoenix, Ariz.

The orderly development of an epitaxial technology requires reliable experimental methods for the measurement of both film thicknesses and internal resistivity gradients. A general review of the possible methods is presented. Special consideration is given to the problem of measuring carrier concentration gradients in directions both normal and parallel to epitaxial growth planes.

Abstract No. 136A

Germanium Epitaxial Deposition in a Resistance Furnace

N. P. Cerniglia and Pei Wang, Semiconductor Div., Sylvania Electric Products Inc., Woburn, Mass.

A technique has been developed using the gas phase deposition of germanium tetrachloride in an open tube resistance furnace for the multislice growth of epitaxial germanium layers. Growth rate and reproducibility, surface finish, and feasibility of the epitaxial layers for device applications have been investigated. The control of growth is dependent on (*a*) vapor transport behavior, (*b*) geometry, (*c*) substrate slice temperature, and (*d*) vapor pressure of germanium tetrachloride.

Abstract No. 137

Surface Effects and Autodoping in Epitaxial Germanium Layers

E. Matovich and **R. J. Andres,** Hughes Products, P. O. Box 278, Newport Beach, Calif.

High-resistivity epitaxial germanium has been grown by using a catalytic reduction of $GeCl_4$ in an open-tube process, at temperatures intermediate to those used for pyrolytic decomposition of $GeCl_4$ (Bell Labs.) and disproportionation of GeI_2 (IBM). Surface structure of, and impurity distribution in, the epitaxial layers are shown to be a function of the growth conditions.

Abstract No. 138

Evaluation of Germanium Epitaxial Films

J. R. Biard and **S. B. Watelski,** Texas Instruments Inc., Box 5012, Dallas 22, Texas

A method has been developed for rapid and accurate determination of the resistivity of p-type epitaxial germanium films deposited on p+ germanium substrates. Determination of the resistivity is not dependent on a knowledge of film thickness. Data are presented for films with resistivities of 0.05-7.0 ohm-cm, deposited on 0.002 ohm-cm substrates; film thicknesses range from 0.15 to 0.6 mil.

Abstract No. 139

Some Properties of Epitaxial Germanium

A. B. Kuper and **H. Christensen,** Bell Telephone Labs., Inc., Murray Hill, N. J.

Germanium (111) epitaxial films have been deposited in an open tube system from H_2-$GeCl_4$ (undoped) at 850°C. Junction capacitance measurements suggest the principal impurity is copper (2.5×10^{14}/cc after 600°C anneal). After etch removal of the substrate one of these films was annealed at 550°C. Sheet resistance measurements as a function of annealing time suggested slow precipitation of copper characteristic of Ge with less than 10^3 dislocations/cm^2. Hall effect hole mobility *vs.* temperature was similar to that in high resistivity melt-grown Ge. Electron transmission diffraction shows no crystalline imperfections in these films.

Abstract No. 140

Vapor Growth of GaAs

V. J. Lyons and **V. J. Silvestri,** Research Center, International Business Machines Corp., Yorktown Heights, N. Y.

It has been demonstrated previously that synthesis and crystal growth of III-V compounds can be carried out at temperatures below the compound melting points by reacting a Group III halide with a Group V element. In this work single crystal films of GaAs have been grown from a vapor phase consisting of gallium iodides and arsenic. The material was deposited epitaxially on monocrystalline GaAs of various crystallographic orientations. The reactions were carried out in sealed quartz tubes under varying conditions of temperature and pressure. Measurements of the resistivity and mobility of the deposited GaAs are presented. Vapor pressure measurements for the system are discussed.

Abstract No. 141

Mechanism for the Vapor Transport of Gallium Arsenide with Hydrogen Chloride

F. V. Williams and **R. A. Ruehrwein,** Research and Engineering Div., Monsanto Chemical Co., Dayton, Ohio

Epitaxial layers of GaAs have been grown on GaAs substrates in an open system using hydrogen chloride

as the transfer agent. The principal features of this process are: (*a*) reaction of GaAs with HCl at temperatures above about 750°C to yield a mixture of gaseous reaction products, and (*b*) reaction of the gaseous reaction products of step (*a*) at lower temperatures to redeposit GaAs. A mechanism for the first step has been deduced. Equilibrium data indicate that a lower chloride of gallium, GaCl, is formed. Thermodynamic constants for the reaction are given. The second step of the process is *not* a simple reversal of the first step. Disproportionation of the lower chloride of gallium occurs, along with redeposition of GaAs, in such a fashion that the theoretical fraction of GaAs which can be redeposited is two-thirds. Some effects of substrate orientation on epitaxial growth are discussed.

Abstract No. 142

An Open Tube Carrier Gas Method for the Epitaxial Growth of Gallium Phosphide

C. J. Frosch and **P. W. Foy,** Bell Telephone Labs., Inc., Murray Hill, N. J.

An open tube processing technique is described for growing epitaxial layers of gallium phosphide. The method is based on an appreciable volatility of a gallium containing vapor species which is believed to be GaP (g). Either undoped or doped epitaxial layers can be grown. Some of the structural and electrical characteristics of these layers are described. Epitaxial layers of gallium arsenide and gallium arsenide phosphide ($GaAs_xP_y$) also can be grown by this technique. It may also prove to be suitable for growing epitaxial layers of other multicomponent semiconductors.

Abstract No. 143

Vapor Phase Growth of GaAs and GaP

R. R. Moest and **B. R. Shupp,** Bell Telephone Labs., Inc., Murray Hill, N. J.

Gallium arsenide and GaP films have been grown in sealed systems charged with HCl gas. The growth rate on GaAs substrates from GaAs sources has been measured as a function of temperature, initial HCl pressure, and substrate orientation. Conductivity type of the films can be controlled by doping the source material. Epitaxial films of GaAs have been grown on Ge and GaP films on GaAs.

Abstract No. 144

The Pseudobinary System Mg_2Si-Mg_2Ge

R. J. LaBotz and **D. R. Mason,** Dept. of Chemical and Metallurgical Engineering, University of Michigan, Ann Arbor, Mich.

Thermal conductivity, electrical conductivity, Hall effect, x-ray diffraction, and differential thermal analysis data have been taken on the pseudobinary system Mg_2Ge—Mg_2Si. The data indicate that Mg_2Ge—Mg_2Si have complete solid solubility, as expected, because of the structural similarity between Mg_2Ge and Mg_2Si. The thermal conductivity, electrical conductivity, and

Hall effect data are analyzed to determine relative variations in the properties as a function of composition.

Abstract No. 145

Impurity Distribution in Single Crystal Indium Antimonide

G. R. Cronin, Texas Instruments Inc., Dallas, Texas

The purpose of this work is to examine the anisotropic segregation of various impurities in InSb. Crystals oriented (111), (110), (311), and (100) were pulled from melts containing radioactive Zn, Sn, Fe, and Ag. Uniformity of distribution is evaluated parallel and perpendicular to the growth direction. The most striking result of this work is the nonuniformity of (111) crystals as compared with other orientations. All of the results are explained in terms of the facet effect.

Abstract No. 146

Distribution of Sulfur Impurity in InSb Single Crystals

M. D. Banus and **H. C. Gatos,** Lincoln Lab., Massachusetts Institute of Technology, Lexington 73, Mass.

Single crystals of InSb doped with inert or radioactive sulfur (δ35) were pulled from the melt using a Czochralski-type technique. The distribution of sulfur was studied by high resolution autoradiography and electrical measurements. In crystals grown along the <111> direction, a well-defined core with increased concentration of sulfur is associated with the {111} facet present at the liquid-solid interface. The effects of changing rotation rate during growth on the concentration "on-facet" and "off-facet" and on the effective distribution coefficients are presented. The results are discussed on the basis of crystal growth mechanisms.

Abstract No. 147

Correlation of Electrical Measurements and Chemical Analyses of Zinc- and Cadmium-Diffused GaAs

J. F. Black, General Telephone and Electronics Labs. Inc., Bayside 60, N. Y.

Thin rectangular slices of single-crystal GaAs were doped to degeneracy by indiffusing zinc or cadmium at 1100°-1200°C from arsenic-metal vapors in evacuated quartz capsules. The gradient of dopant concentration throughout the thickness of the slices was controlled over a wide range. In several cases the dopant concentration at the median plane of the slice was as an order of magnitude lower than at the surface of the slice. In all instances, however, the free hole concentration calculated from Hall effect measurements agreed with the average dopant concentration determined by chemical analysis. Data on measured Hall mobilities are presented and compared with data for melt-doped GaAs.

Abstract No. 148

Correlation of Lattice Parameter Measurements and Electrical Measurements in Highly Doped GaAs

J. F. Black and **P. Lublin,** General Telephone and Electronics Labs., Inc., Bayside 60, N. Y.

The variation in the lattice parameter of GaAs was studied as a function of doping with donor and acceptor elements, including Cd, Zn, Te, and Se. A modified precision lattice constant technique for powders was used; this can detect changes in the lattice constant of ±0.0002Å in GaAs. On the basis of these lattice parameter measurements the solubility of the dopants could be determined. Data on Hall effect and resistivity are presented and correlated with the x-ray data.

Abstract No. 149

Nondendritic Ribbon Crystals

H. Norman, G. Russell, and **M. Forney,** Semiconductor Products Div., Motorola, 5005 E. McDowell Rd., Phoenix, Ariz.

A technique for growing single crystals of germanium by a nondendritic process is presented. The dependence of the process on crystallographic orientation of the seed and a restricted meniscus is discussed. The necessary temperature gradients to allow feasible growth rates are described and techniques for maintaining these gradients discussed.

Abstract No. 150

Preparation of High-Purity Silicon from Silane

C. H. Lewis, M. B. Giusto, H. C. Kelly, and **Sidney Johnson,** Metal Hydrides Inc., Beverly, Mass.

The preparation, purification, and thermal decomposition of silane, SiH_4, were investigated for the purpose of preparing high-purity silicon. Quantitative yields of silane were obtained by the reduction of silicon tetrachloride with various hydrides. The reactivity of silane with various purifying agents was studied. Variables in the thermal decomposition of silane were evaluated with regard to purity of the elemental silicon produced, and the efficiency of dissociation and deposition rate. Silane was dissociated on an inductively heated single crystal silicon substrate to yield high-purity silicon. Neutron activation analyses for microcrystalline high-purity silicon as deposited are indicated below:

Impurities in Silicon Metal from Thermal Decomposition of Silane

Neutron activation analyses (ppb)

Fe	17.0*	As	1.0	Zn	0.41*	Ga	0.0062*
Cu	11.0	P	1.0*	Ni	0.27*	Sb	0.0037*
Bi	1.6*	Tl	0.91*	In	0.032*	Mn	0.00062*

* Not detected. Value shown is limit of detection in ppb.

Abstract No. 151

The Effect of Heat Treatment, Oxygen, and Structure on the Lifetime of Float Zoned Silicon Crystals

M. D. Jordan and **E. W. Stewart,** E. I. du Pont de Nemours & Co., Brevard, N. C.

Lifetime of float zoned silicon crystals is shown to be independent of oxygen content, crystal structure, and initial lifetime values after heat treatment at diffusion temperatures. Samples have been analyzed by copper decoration techniques and neutron activation in an attempt to explain these results. The data obtained are compared to previous work, and the effect of these phenomena on device production are postulated.

Abstract No. 152

Measurement Errors of Silicon Single Crystals and Slices

J. S. Miller, and **V. E. Paulos,** Materials and Sensors Quality Assurance, Texas Instruments Inc., P. O. Box 312, Dallas 21, Texas

One of the most important aspects connected with the measurement of any parameter for practically all products is the amount of error involved when making such a measurement. If this error is not determined or estimated, then the accuracy and precision of the measurement is unknown. The use of such information, when the error is unknown, could be extremely expensive by causing excessive rejection of products which otherwise might have been acceptable. This is especially true of silicon single crystals and slices used in the manufacture of semiconductor devices.

At Texas Instruments, there are many parameters which are used to determine the quality of silicon single crystals and slices such as the grade of silicon used, its boron and phosphorus content, the lifetime of the crystal, dislocation density, diameter, resistivity, general appearance, etc. However, for the purpose of this discussion we will deal only with crystal and slice resistivity and slice thickness.

Abstract No. 153

Resistivity Measurements at Microwave Frequencies

G. L. Allerton and **J. R. Seifert,** Western Electric Co., 555 Union Blvd., Allentown, Pa.

Resistivity measurements can be made at microwave frequencies with less damage to the material than obtained by a conventional four point probe and in a faster and more accurate manner. A high Q resonant circuit has been developed for low resistivity ranges of 0.002 to 1.0 ohm-cm. A second method of measuring the transmission loss through thin slices is used to cover the resistivity range from 0.1 to 40 ohm-cm.

Abstract No. 154

Silicon Crystal Perfection Measurement and Grading

M. R. Everingham, E. I. du Pont de Nemours & Co., Inc., Brevard, N. C.

Several sample preparation and etching techniques to reveal structural defects in silicon crystals are compared. One method is described which provides excellent reproducibility in a reasonable period of time. A system of numerical grading to designate the relative severity of lineage and slip is outlined. This system has proven useful in standardizing perfection measurements between various laboratories.

Abstract No. 155

Horizontal Zone Leveling of Silicon

J. L. Porter, Sylvania Electric Products Inc., Woburn, Mass., and **D. M. Lamb,** Sylvania Electric Products Inc., Towanda, Pa.

A method for the horizontal zone leveling of silicon is described. Maintaining the walls of the vessel at a temperature below the melting point of silicon prevents adhesion of the silicon upon solidification. Such a technique facilitates the formation of large, heavily doped silicon crystals for microwave application.

Abstract No. 156

Iron-Boron Interactions in Silicon

W. H. Shepherd and **J. Turner,** Research Labs., The Plessey Co. Ltd., Caswell, Towcester, Northants, England

Time dependent resistivity changes in p-type silicon, saturated with iron and quenched from above 900°C, are shown to be due to pairing between iron and boron. At 45°C more than 50% of the boron may be paired. Precipitation of the iron has also been studied. Below 200°C the iron solubility after precipitation is strongly influenced by pairing and electron hole equilibria. Analysis of these data gives the true solubility of iron at these temperatures.

Abstract No. 157

Trace-Plating

W. Rindner and **J. M. Lavine,** Research Div., Raytheon Co., Waltham, Mass.

The observation of nucleation of plated metals on lines scribed on semiconductors using hard stylus materials had led to investigations showing that preferential plating can also be obtained on thin films traced with soft styli on semiconducting and insulating materials. Trace and plating materials may be either doping elements or carrier metals. Ohmic and rectifying contacts to Ge and Si as well as mesa transistors have been fabricated by trace-plating in conjunction with conventional alloying techniques.

ELECTRO-ORGANIC

Abstract No. 158

Improvement in the Electrolytic Preparation of Iodoform

R. Ramaswamy, M. S. Venkatachalapathy, and **H. V. K. Udupa,** Central Electrochemical Research Institute, Karaikudi 3, S. Rly., India

Use of a lead dioxide anode with a graphite substrate in the electrolytic preparation of iodoform is described. Influence of bath composition, temperature, and pH on the current efficiency has been investigated. Results are compared with those obtained with the conventional anodes like platinum and graphite. Effect of rotation of the anode in the preparation is discussed.

Abstract No. 159

Electrolytically Regenerated Manganic Sulfate for the Oxidation of Aromatic Hydrocarbons, II. Oxidation of Xylenes to Tolualdehydes

R. Ramaswamy, M. S. Venkatachalapathy, and **H. V. K. Udupa,** Central Electrochemical Research Institute, Karaikudi 3, S. Rly., India

The electrolytic oxidation of p-xylene to p-tolualdehyde has been investigated both by a single stage process as well as by a two-stage process using lead dioxide anode. The single stage process using manganous sulfate as "oxygen carrier" gave a low current efficiency (<30%), whereas the two-stage process gave an overall current efficiency of 40-45%. Manganous sulfate as a suspension in 55% H_2SO_4 could be oxidized anodically to a manganic sulfate paste which could be used in the controlled oxidation of xylenes to tolualdehydes. Results obtained in this cyclic process are presented.

Abstract No. 160

Stereochemistry of Electrolytic Reductions of Butynediol and Cyclohexene- 1,2- Dicarboxylic Acid

George Brownell and **C. L. Wilson,** Phillips-Foscue Corp., Box 1306, High Point, N. C.

(No abstract received)

Abstract No. 161

Electrolytic Formation of Oximes

Tadao Hayashi, Dept. of Applied Chemistry, University of Osaka Prefecture, Mozu-Umeno-cho, Sakai City, Osaka, Japan and **C. L. Wilson,** Phillips-Foscue Corp., Box 1306, High Point, N. C.

(No abstract received)

Abstract No. 162

New Study on the Electrolytic Reduction of Dicyandiamide. An Approach to the Essential Problem on Organic Electrode Process

Kiichiro Sugino, Keijiro Odo, and **Eiichi Ichikawa,** Tokyo Institute of Technology, Ookayama Meguro-ku, Tokyo, Japan

Although dicyandianide gave no distractive polarographic wave, it was reduced at a mercury cathode at —1.05 v (*vs.* S.C.E.) to give a 4 hydrogen addition compound; at a palladium-black cathode dicyandiamide was reduced at —0.10 v (*vs.* S.C.E.) with 100% current efficiency to give a 2 hydrogen addition compound. Thus the observed difference in reduction potentials and products indicates the essential difference between reduction by direct electron transfer (Hg electrode) and reduction by chemisorbed hydrogen (Pd-black electrode).

Abstract No. 163

A Continuous Flow Cell for Electrochemical Synthesis

M. J. Allen, Chemical Research Dept., Electro-Optical Systems, Inc., Pasadena, Calif.

One of the major drawbacks to the use of electrochemical techniques in the production of synthetic organic chemicals is the unavailability of an efficient, readily fabricated continuous flow cell. A pilot plant cell has been developed which consists of thin polysterene compartments. The odd numbered compartments are separated from the next higher even numbered compartments by an ion-exchange membrane. Located between the even numbered compartments and the next higher odd numbered compartments are floating electrodes. The terminal electrodes at each end of the cell are connected to a d-c source. Each compartment has an inlet and outlet through which the catholyte and anolyte flow into alternate cell sections. A study of the reduction of p-hydroxybenzaldehyde has been made utilizing this cell.

Abstract No. 164

Voltammetric Study of the Kolbe Reaction

M. J. Allen, Chemical Research Dept., Electro-Optical Systems, Inc., Pasadena, Calif., and **W. W. Pierson,** Dept. of Chemistry, Seton Hall University, South Orange, N. J.

Various acids such as acetic, caproic, heptanoic, isocaproic, isovaleric, octanoic, and valeric acid were investigated as their sodium salt in a methanolic medium at a rotating platinum anode. The data obtained are discussed and correlated with free radical formation at the electrode surface. A comparison is also made with the data obtained on sodium acetylglycinate, which is known not to undergo the typical Kolbe dimerization.

Abstract No. 165

Anhydrous Voltammetry of the Acetate Ion at a Rotating Platinum Anode

M. J. Allen, Chemical Research Dept., Electro-Optical Systems, Inc., Pasadena, Calif., and **W. W. Pierson,** Dept. of Chemistry, Seton Hall University, South Orange, N. J.

The most commonly accepted theory on the mechanism of the Kolbe reaction is the discharged ion theory of Brown and Walker whereby the acetate loses one electron to the anode resulting in an acetate radical which spontaneously splits off CO_2 and produces a methyl radical which is free to react with a second methyl radical to give the Kolbe dimer, ethane. If this theory is correct, the discharge of acetate ion should give an irreversible voltammetric step where the diffusion current varies linearly with acetate ion concentration. The acetate ion was investigated in a methanolic medium in concentrations of from 0.08M to 1.0M. The half-wave potentials (corr.) *vs.* Ag, AgCl electrode varied from +1.64 v for the lower concentration to +1.25 v for the higher concentration. The diffusion current varied linearly from 0.32 to 3.28 ma.

Abstract No. 166

Electrode Processes in Acetonitrile

D. H. Geske, Dept. of Chemistry, Cornell University, Ithaca, N. Y.

The properties of acetonitrile make it a uniquely suitable media for the investigation of organic electrode processes. In particular, resistance of the solvent itself to electro-oxidation is important. Recent electrochemical investigations are discussed.

Abstract No. 167

The Role of Proton Addition in the Polarographic Reduction of Aromatic Hydrocarbons and Carbonyl Compounds in Dimethylformamide

P. H. Given, Pennsylvania State University, University Park, Pa.

The electrochemical reduction of a neutral molecule requires addition of protons as well as electrons, and proton addition may well be irreversible. By working in a suitable organic solvent, protonation can be made so slow that electron addition by itself can be studied. The addition of weak acids to solutions in such solvents alters the polarographic behavior in a variety of ways, which throw light on polarographic mechanisms; phenol is particularly useful as a protonating agent since it is not reducible at the dropping mercury electrode within the accessible range of potentials, nor does it give a hydrogen discharge wave in dimethylformamide. Experimental data for several systems are discussed to illustrate the above generalities. Evidence is presented

that in dimethylformamide the effective protonating agent is the undissociated acid and not the solvated proton.

Abstract No. 168

Polarography and Voltammetry in Dimethylsulfoxide

I. M. Kolthoff and **T. B. Reddy,** (present address: Bell Telephone Labs. Inc., Murray Hill, N. J.) University of Minnesota, Minneapolis 14, Minn.

A polarographic study of several acids, some representative metal ions, a few quinones and oxygen has been made in the solvent dimethylsulfoxide (DMSO). Perchloric, sulfuric, and hydrochloric acids were found to be strong, monoprotic acids in DMSO. Cobalt(II) and nickel(II) are reduced irreversibly at the dropping mercury electrode with large overpotentials indicating strong solvation. Polarograms of quinones gave two cathodic waves of unequal height in the absence of proton donor. The effect of proton donor on the polarograms of quinones was studied. The mechanism of the reduction of quinones in DMSO is discussed. A rotated platinum and rotated mercury pool electrodes can be used to advantage in DMSO.

Abstract No. 169

Voltammetry in Liquid Sulfur Dioxide

P. J. Elving and **Elio Vianello,** Dept. of Chemistry, University of Michigan, Ann Arbor, Mich.

Although numerous excellent studies of voltammetry in nonaqueous solvents and, more particularly, in nonprotonic-releasing solvents have been reported, liquid sulfur dioxide is apparently the first nonprotonic solvent to be investigated as a medium for voltammetry. Sulfur dioxide has an interesting possible advantage in investigating the mechanisms of organic electrode reactions because of its effectiveness as a reaction medium in stabilizing free radicals. The experimental principles of voltammetry in sulfur dioxide are described: use of vacuum line technique for preparing solutions and maintaining them during measurement, usable indicating and reference electrodes, and available background electrolytes. Attention is given to the implication of the possible reaction of the solvent with indicating and reference electrodes and the minimization of the solution resistance. Results obtained in the cathodic and anodic voltammetry of various inorganic and organic depolarizers are discussed.

Abstract No. 170

The Uses of Electrical Methods in Organometal Systems

R. E. Dessy, Rudolf Salinger, and **Richard Jones,** Dept. of Chemistry, University of Cincinnati, Cincinnati, Ohio

During recent years considerable effort has been expanded on the elucidation of the structure of the Grignard reagent, "RMgX," resulting from the reaction of RX and Mg in ether. A combination of tracer techniques and electrolysis experiments, along with conductivity experiments, indicates that there are two distinct types of Mg in the compound, and that an empirical formula R_2Mg, MgX_2 is a better representation than RMgX. Recent kinetic investigations on the reactions with Schiff bases involving measurement of dielectric constant show that two distinct types of R group are present during reaction, confirming the above hypothesis.

Abstract No. 171

Electrochemical Preparation of Polyfluoro Chemicals in Anhydrous HF

R. D. Dresdner, University of Florida, Gainesville, Fla.

Many organic materials are ionogens in anhydrous HF providing conducting solutions. When a direct current at (4-6 v) voltages is passed through such solutions, hydrogen is evolved at the cathode and a myriad of products containing fluorine are found. In many of these the hydrogen has been completely replaced by fluorine. Organic materials containing groups such as carboxyl, acyl halide, sulfonyl halide, ether, sulfide, amine, and sulfoxide lend themselves to this process to varying degrees. The products formed in the process depend to a large extent on the many variables that require a degree of control, but because of the high energies involved, structure retention is the frequent exception rather than the rule. Fragmentation, cyclization of acyclic starting materials, acyclization of branched starting material, and polymerization are encountered along with a high degree of fluorination of the starting structures. Elements in the products are generally found in their highest oxidation states. The process is still an art in many respects and any degree of predictability of products is still a hazard. This paper attempts to review the results of the current literature in the field.

Abstract No. 172

Polarographic Studies in Acetonitrile and Dimethylformamide, VII. Behavior of Benzyl Halides and Related Compounds

S. Wawzonek and **R. C. Duty,** Dept. of Chemistry, State University of Iowa, Iowa City, Iowa

Polarographic studies of benzyl chloride, benzyl bromide, benzyl iodide, benzylmercuric iodide, dibenzylmercury, mercuric iodide, ethyl chloracetate and ethyl bromoacetate have been made in acetonitrile and dimethylformamide containing tetrabutylammonium salts. Benzyl chloride and ethyl chloracetate were found to undergo a facile displacement reaction with bromide ion. Benzyl chloride and bromide react with iodide ion to form benzyl iodide which reacts rapidly with mer-

cury and forms benzylmercuric iodide. The latter compound, upon standing, slowly forms mercuric iodide.

Abstract No. 173

Polarographic Reduction of Some Diketones and Pyrones

R. L. Flurry, R. H. Philip, and **R. A. Day, Jr.,** Dept. of Chemistry, Emory University, Atlanta 22, Ga.

Additional studies on the polarographic reduction of benzil, benzoin, and several diketones have been carried out. A mechanism is proposed to account for the differences in the ease of rearrangement of the intermediate dienol assumed to be formed in the reduction of the diketone. This ease of rearrangement is related to the pH at which the second wave is observed for certain diketones. Preliminary results on the reduction characteristics of γ-pyrone and several substituted pyrones are reported.

Abstract No. 174

Electrochemical Reduction of the Benzene Ring

H. W. Sternberg, Raymond Markby, and **Irving Wender,** U.S. Dept. of the Interior, Bureau of Mines, Pittsburgh 13, Pa.

Application of electrolytic methods to coal chemistry has been restricted by the fact that the benzene ring, which represents a large portion of the coal structure, could not be reduced electrolytically. We now have found that electrolysis of tetralin in methylamine saturated with lithium chloride gives hexalin and octalin. The possibility of direct electron transfer from cathode to tetralin is discussed, and the implications of this reaction for the polarographic determination and electrolytic reduction of the benzene ring are pointed out.

Abstract No. 175

Kolbe and Crum Brown-Walker Syntheses with Perfluoroaliphatic Acids

Sherlock Swann, Jr., and **F. H. Baastad** (Fellow of the Royal Norwegian Council for Scientific and Industrial Research, 1952-53, Present address: Finn Baastad, Grandeveien 5, Bygdoy, Oslo, Norway), Dept. of Chemistry and Chemical Engineering, University of Illinois, Urbana, Ill.

The Kolbe electrolytic synthesis of tetradecafluorohexane from free heptafluorobutyric acid has been carried out in excellent yield. It has been found possible to prepare the half fluorinated hexane by electrolysis of a mixture of the potassium salts of butyric acid and heptafluorobutyric acid. Similarly, ethyl heptafluorododecanoate has been obtained from the potassium salts of monoethyl sebacate and heptafluorobutyric acid. Electrolyses of the potassium salts of nonanoic and pentadecafluoroöctanoic acids have also been carried out.

Abstract No. 176

Anodic Oxidation in a Heterogeneous System

H. F. Conway, E. B. Lancaster, V. E. Sohns, and **S. Dabic,** Northern Regional Research Lab., Peoria, Ill. (This is a Laboratory of the Northern Utilization Research and Development Div., Agricultural Research Service, U.S. Dept. of Agriculture)

In-cell oxidation of starch to dialdehyde starch by electrolytically generated periodic acid is a pseudo electro-organic reaction. Possible reactions of starch and dialdehyde starch with components of the reaction mixture under conditions in the cell are reviewed. The mechanical features of the cell that can influence starch oxidation and the engineering problems encountered in the initial pilot-scale production are discussed.

Abstract No. 177

Experimental Study of the Anode Effect in the Electrolysis of Fused Mixtures of Sodium Chloride and Zirconium Tetrafluoride

C. M. Decroly and **René Winand,** Lab. of Metallurgy and Electrochemistry, University of Brussels, 50 Ave. F.-D. Roosevelt, Brussels 5, Belgium

An experimental investigation has been made of the anode effect when electrolyzing mixtures of fused sodium chloride and zirconium tetrafluoride. Factors which have been studied were: composition of the melt, diameter of the graphite cylindrical anode, temperature, speed of rotation of the anode, and addition of zirconium dioxide. It can be shown that there exists a critical current density under which it is impossible to observe any anode effect. At a given temperature, these critical current densities depend on the composition of the melt and on the diameter of the anode. No marked effect of temperature, rotation of the anode, and addition of zirconium dioxide could be observed. A preliminary explanation of the facts observed is given.

Abstract No. 178

Ionic Transference in Pure Molten Sodium Nitrate

R. J. Labrie and **V. A. Lamb,** Metallurgy Div., National Bureau of Standards, Washington 25, D. C.

Transference numbers of sodium and nitrate ions in pure molten sodium nitrate have been measured in "H-shaped" cells containing a porous diaphragm between anode and cathode compartments. The cells differ from those used in previous researches in that legs made of sodium-ion-conductive porcelain served to carry the current of sodium ions into and out of the cell. This design allows repeated observations with high experimental precision. Transference number values depended on the material of the diaphragm. Diaphragms were of porcelain, alundum, and fritted Pyrex.

ELECTROTHERMICS AND METALLURGY

Abstract No. 179

Progress in Sintering

D. R. Wilder, Ames Lab., Iowa State University of Science and Technology, Ames, Iowa

(No abstract received)

Abstract No. 180

Some Effects of Powder Properties and Consolidation Technique on the Properties of Sintered Tungster Compacts

G. C. Bodine, Fansteel Metallurgical Corp., North Chicago, Ill.

The initial phase of the Bureau of Naval Weapons Tungsten Sheet Rolling Program included the evaluation of candidate tungsten powders with respect to the effect of particle distribution, density, and chemistry on consolidation to sintered billets. The relationship between compaction pressure and sintering cycle was also studied. The results of this investigation are presented.

Abstract No. 181

Activated Sintering of Tungsten

J. H. Brophy, H. W. Hayden, and **J. Wulff,** Dept. of Metallurgy, Massachusetts Institute of Technology, Cambridge 39, Mass.

Small additions of nickel to tungsten permit the strengthening and densification of both powder compacts and plasma-sprayed deposits at relatively low temperatures while maintaining a relatively high melting point. Theoretically, the mechanism appears to be a kinetic intermediate between current theories of solid and liquid phase sintering. Several other activating agents have been employed successfully, and in each case the densification is kinetically similar.

Abstract No. 182

Sintering Characteristics of Ultrafine Tungsten Powder

C. R. McKinsey, Union Carbide Metals Co., Niagara Falls, N. Y.

(No abstract received)

Abstract No. 183

4th Plansee Seminar

H. A. Johansen, Research Labs., Westinghouse Electric Co., Beulah Rd., Churchill Boro., Pittsburgh 35, Pa.

(No abstract received)

Abstract No. 184

Sintering of Pressureless Compacted Tungsten and Molybdenum

H. H. Hausner, Adjunct Professor, Polytechnic Institute of Brooklyn, and Consulting Engineer, 730 5th Ave., New York 19, N. Y., and **A. R. Poster,** Sylvania Electric Products Inc., Towanda, Pa.

The densification rate during sintering of a mass of powder can be expressed in an equation consisting of three components, dealing with the density before sintering, powder particle size, and particle activity. The methods of sintering tungsten and molybdenum powders compacted under pressure are well known. These powders, however, can be sintered also as loose particles or as slip cast, and the rate of sintering in this case depends to a large extent on the particle characteristics. The "activity" of the powder particles is discussed. Surprisingly high densities of tungsten and molybdenum can be obtained by sintering these pressureless compacted powders. Explanation of this effect is given.

Abstract No. 184A

Sintering of Slip Cast Tungsten

F. R. Charvat, Union Carbide Metals Co., 4625 Royal Ave., Niagara Falls, N. Y. and **G. Reinhardt,** National Aeronautics and Space Administration, Lewis Research Center, Cleveland 35, Ohio

The effect of sintering variables including temperature in the range 1500°-2250°C, and time from 0.5 to 4 hr with vacuum and hydrogen atmosphere are characterized for slip-cast tungsten. The results of the analysis are presented graphically with densification parameter as dependent variable. The use of the curves to calculate linear shrinkage and density is illustrated. An indication of the degree to which dimensional tolerances may be controlled in the slip-casting and sintering processes is obtained by statistical analysis of green and sintered dimensions.

Abstract No. 185

Plasma Spraying of Tungsten and Refractory Compounds and the Influence of Sintering on the Physical Properties of These Materials

Irving Strauss and **Robert Steinitz,** General Telephone & Electronics Labs. Inc., Bayside 60, N. Y.

The preparation of parts from tungsten and refractory compounds, such as tantalum carbide, by plasma spraying is described. The behavior of the deposited material during sintering at various temperatures is studied and compared with that of the same material produced by standard powder metallurgy techniques. The rate of densification of the sprayed material during sintering is compared with that of cold pressed material, and the influence of sintering on some physical prop-

erties, such as strength, microstructure, and electrical resistivity, is reported.

Abstract No. 186

Preparation of F48 Alloy by Means of Hydrostatic Pressing and Vacuum Sintering

J. W. Pugh and **J. F. Cerness,** Lamp Metals and Components Dept., General Electric Co., 21800 Tungsten Rd., Cleveland 17, Ohio

The experiments described were designed to prove the feasibility of the powder metallurgy approach to the manufacture of F48 Alloy (Cb-15W 5Mo 1Zr). Both pre-alloyed powders and elemental blends were hydrostatically pressed and vacuum sintered. The effect of sintering time and temperature on density, grain size, purity, hardness, and homogeneity was evaluated and analyzed. A two step sintering cycle was designed, and experimental samples were made and tested for fabricability by forging and rolling at room temperature as well as at elevated temperatures. Sheet made from these samples was evaluated for recrystallization temperature, hardness, purity, microstructure, and tensile properties both at room temperature and at 2200°F. Results indicate that F48 billets produced by this method are more fabricable than those produced by arc-casting. Properties of the sheet produced were generally equivalent to those of sheet made by arc-casting, except that the tensile strength was slightly higher and the elongation was usually lower. These deviations are attributed to differences in purity.

Abstract No. 187

Sintering of Oxides

G. C. Kuczynski, Metallurgy Dept., University of Notre Dame, Notre Dame, Ind.

(No abstract received)

Abstract No. 188

A Study of the Sintering Behavior of Some Uranium Dioxide Powders

J. C. Clayton and **L. Berrin,** Bettis Atomic Power Lab., Westinghouse Electric Corp., Pittsburgh 30, Pa.

The sintering behavior of some 35 well-characterized uranium dioxide powders was studied. Pressing pressures and sintering time, temperature, and atmosphere were varied. Definite relationships were found between the sinterability of the powders and their preparation method, surface texture, particle size and shape, and aggregate size and distribution.

Abstract No. 189

High-Temperature Electrical Conduction in the System UO_2-ZrO_2

H. A. Johansen and **J. G. Cleary,** Research Labs., Westinghouse Electric Corp., Beulah Rd., Churchill Boro., Pittsburgh 35, Pa.

Present knowledge of the phase relationships and electrical properties is briefly reviewed and discussed. Examination is made of the dependence of the electrical conduction on density, composition, temperature, and atmosphere. The nature of the electrical conduction is investigated.

Abstract No. 190

Sintering Kinetics of Linde A Al_2O_3

C. A. Bruch, Research Lab., General Electric Co., Schenectady, N. Y.

(No abstract received)

Abstract No. 191

Sintering of Hafnium Carbide with a Fugitive Metal Binder

Charles Zalabak, NASA, Lewis Research Center, Cleveland, Ohio

Hafnium carbide powder mechanically mixed with cobalt powder has been cold compacted and sintered in such manner as to remove the cobalt by vaporization. Densities of 95% of theoretical have been achieved with surface connected porosity of about 2%. Observations are made of the variations in grain size and microhardness obtained over the range of process variables. The variables and range covered were: added cobalt, 0.5 to 5% by weight; maximum temperature, 3500° to 4500°F; time at maximum temperature, 1/10 and 1/2 hr; and heating cycle.

Abstract No. 192

Sintering of Pure SiC

G. R. Watson and **J. I. Frederiksson,** Norton Co., Niagara Falls, Canada

The process of manufacture, properties, and uses of a new slip-cast pure SiC refractory (U.S. Pat. 2,964,823) are described. Previous attempts to make such bodies failed. The finished articles can be made with controlled porosities between about 20 and 50% and may be permeable or impermeable to fluids, as desired. The physical properties for representative types are illustrated. Practical applications include: rocket nozzle throats, permeable filters for corrosive gases or liquids, resistors, and various refractory specialties.

Abstract No. 193

Furnaces, Supports, and Atmospheres for the Sintering of Some Borides, Nitrides, and Silicides

A. Blum, Borolite Corp., 3113 Forbes Ave., Pittsburgh 30, Pa.

In the sintering of borides, nitrides, and silicides of high purity, furnace equipment, supports, and the atmosphere must be chosen so as to minimize contamination of the charge during the sintering process. This choice must be made on the basis of the thermodynamic properties of the respective materials as well as of the kinetics of the reaction between the charge and the materials with which it may be in contact. Illustrations of the sintering conditions for some borides, nitrides, and silicides are given.

Abstract No. 194

The Sintering of Dense, Fine-Grained Beryllides

R. S. Truesdale, B. B. Lympany, and **W. W. Beaver,** The Brush Beryllium Co., Cleveland, Ohio

Fabrication of the intermetallic compounds of beryllium is normally accomplished through hot pressing or sintering. The standard ceramic process of cold pressing and sintering has been successfully applied to these materials. The results of sintering studies on the compound Nb_2Be_{17} and other beryllide compounds are presented. The effect of particle size, stoichiometry, sintering atmosphere, temperature, time on density, grain size, and modulus of rupture of the materials are described.

Abstract No. 195

Electrochemical Oxidation of Titanium Surfaces

M. E. Sibert, Missiles and Space Div., Lockheed Aircraft Corp., Palo Alto, Calif.

Films produced on titanium depend on nature and concentration of electrolyte, forming voltage, current density, and temperature. In most cases, dense films of limited thickness are produced, but in some cases thick porous films result. Physical, chemical, and electrical nature of the coatings are discussed, together with their relation to the surface character. Evidence indicates that films prepared in aqueous electrolytes are not of the rutile structure. These coatings are highly uniform, insulating, and adherent.

Abstract No. 196

Anodic Formation of Titanium Oxide Dielectric Films

R. T. Lamoureux, Missiles and Space Div., Lockheed Aircraft Corp., Palo Alto, Calif.

Equipment is described which was employed for continuous measurement of dielectric properties of electrochemically produced films on titanium sheet. Data obtained using this equipment are discussed in relation to the process parameters of dielectric film formation. Effects of time, temperature, and humidity on the titanium oxide dielectrics prepared are also described.

Abstract No. 197

A Study of the Anodic Film Formed on Titanium

M. Tecotzky, Missiles and Space Div., Lockheed Aircraft Corp., Palo Alto, Calif.

A physical and chemical study has been made of the anodic film formed on titanium. The thin films studied were prepared by anodizing titanium in an aqueous solution of monoethyl and diethyl hydrogen phosphate, A study was made of film thickness. The physical and chemical form of the film was examined using x-ray and electron diffraction techniques, as well as chemical investigation.

Abstract No. 198

Silicon Carbide—An Electrolytic Capacitor Material

B. J. Nicholson, W. Haas, and **J. J. Flood,** Research Div., Philco Corp., Blue Bell, Pa.

Solid electrolytic capacitors have been developed which have for dielectric a thin film of silica grown by thermal oxidation on silicon carbide. Use of a silicon carbide foam permits construction of units with large capacity per unit volume. After oxidation, the foam is anodically "formed" in an electrolyte of KNO_3 in N-methylacetamide; this is only possible when impurities in the foam are very small. MNO_2 counterelectrodes are then applied. Parameters of these capacitors are described.

Abstract No. 199

Characterization and Properties of UC Crystals Produced by Means of Electron-Beam Zone Melting

Roger Chang and **Harry Nadler,** Atomics International, Div. of North American Aviation, Inc., Canoga Park, Calif.

The preparation of UC single crystals in dimensions up to 10 cm in length and ½ cm in diameter by means of electron beam zone-melting is described. Orientation control was accomplished by seeding. The zone-melted crystals were slightly hyperstoichiometric in C, and methods of removal of the excess C are presented. Reduction in impurities by zone-refining and microstructural studies of the nature of imperfections in UC crystals made by this technique are also reported. (This work was supported by the U.S.A.E.C.)

Abstract No. 200

Mechanical Properties of Nearly Stoichiometric UC Polycrystals between 1500° and 2000°C

Roger Chang, Atomics International, Div. of North American Aviation, Inc., Canoga Park, Calif.

The creep, flow, and recovery behavior of nearly stoichiometric arc-cast polycrystalline UC in the temperature range 1500°-2000°C is reported. Deformation of UC in this temperature range is shown to be diffusion-controlled and has almost all the characteristics of a metal. Analysis of the experimental data in terms of current dislocation theories is also presented. (This work was supported by the U.S.A.E.C.)

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INDEX TO AUTHORS

(The number in the right-hand column is the abstract number used in the Technical Program and in the Abstracts)

	Abstract No.
Agruss, B.	38, 40
Albers, W. A., Jr.	113
Alexander, Rexford	95
Allen, M. J.	163, 164, 165
Allerton, G. L.	153
Andres, R. J.	137
Atalla, M. M.	135
Atkinson, J. T. N.	66
Baasted, F. H.	175
Bagrowski, J.	110
Bakish, R.	60, 63
Banus, M. D.	146
Barak, M.	22
Barnes, S. C.	71, 80
Bartlett, J. H.	65
Bate, G.	101
Beach, J. G.	93
Beacom, S. E.	81, 91
Beaver, W. W.	194
Beck, A. F.	58
Beck, Walter	112
Bellobono, I. R.	89
Benjamin, C. E.	120
Bennett, R. J.	31
Berger, Carl	28, 29
Bergh, A. A.	125
Berrin, L.	188
Bertorelle, Eugenio	72, 89
Biard, J. R.	138
Bicelli, L. P.	84
Bishop, C. R.	50
Black, J. F.	147, 148
Blum, A.	193
Boddy, P. J.	114
Bodine, G. C.	180
Bond, A. P.	19
Booker, G. R.	120
Borders, E.	60, 63
Brattain, W. H.	114
Bregman, J. I.	27
Brewer, E. G.	64
Brophy, J. H.	181
Brown, H.	69
Brownell, George	160
Bruch, C. A.	190
Buck, R. P.	18
Bundy, W. S.	13
Busen, Karl	124
Bush, G. W.	74

Abstract No.

Cahill, A. E. 107
Cahoon, N. C. 1
Carr, J. M. 103, 111
Cerness, J. F. 186
Cerniglia, N. P. 136A
Chang, Roger 199, 200
Charvat, F. R. 184A
Chilton, W. H. 109
Chludzinski, P. 26
Christensen, H. 139
Clayton, J. C. 188
Cleary, J. G. 189
Clemson, D. 96
Cohen, M. 64
Conway, B. E. 70
Conway, H. F. 176
Cook, H. C. 75
Cronin, G. R. 145
Cubiciotti, Daniel 32

Dabic, S. 176
Daley, J. L. S. 4
Day, R. A., Jr. 173
Deal, B. E. 133
DeBoer, F. E. 48
Decroly, C. M. 177
Dessy, R. E. 170
DeVan, J. H. 36
Dirkse, T. P. 6
Draley, J. E. 48
Dravnieks, Andrew 27
Dresdner, R. D. 171
Duddy, J. C. 8
Duty, R. C. 172

Eicke, W. G., Jr. 9
Elving, P. J. 169
Ervin, Guy, Jr. 52
Evans, S. 47
Everingham, M. R. 154

Farr, J. P. G. 71, 79
Felten, E. J. 55
Fickett, A. P. 26
Fisher, R. D. 104, 109
Flood, J. J. 198
Flurry, R. L. 173
Foley, M. A. 108
Forney, M. 149
Foulke, G. D. 85
Foy, P. W. 142
Frankl, D. R. 121
Franklin, T. C. 92
Fredriksson, J. I. 192
Frosch, C. J. 142

Gatos, H. C. 146
Geske, D. H. 166
Gillibrand, M. I. 22
Ginell, W. S. 37

Abstract No.

Giusto, M. B. 150
Given, P. H. 167
Glaser, J. H. 100
Grantham, L. F. 37
Gray, J. 22
Greenberg, Sherman 53
Greenblatt, J. H. 66
Greene, N. D. 50, 61
Greig, W. J. 116
Griffith, L. R. 18

Haas, W. 198
Hackerman, Norman 45, 68
Harris, W. S. 2
Hausner, H. H. 184
Hayashi, Tadao 90, 161
Hayden, H. W. 181
Heath, C. W. 129
Hietbrink, E. H. 40
Hiller, M. A. 37
Hills, Stanley 5
Hino, K. 59
Hoar, T. P. 76
Hoekstra, J. J. 86
Hollyer, R. N., Jr. 81
Hovorka, Frank 24, 88
Howard, P. L. 7
Hunger, H. F. 16

Icenhower, D. E. 19
Ichikawa, Eiichi 162
Indig, M. E. 41
Ishida, Takeo 90

James, W. J. 56
Jankowsky, E. J. 112
Johansen, H. A. 183, 189
Johnson, A. A. 94
Johnson, G. R. 82, 83
Johnson, J. W. 56
Johnson, Sidney 150
Jones, E. L. 11, 12
Jones, Richard 170
Jordan, M. D. 151

Kahng, D. 135
Kass, S. 49
Keen, J. M. 79
Kelly, H. C. 150
Koehler, E. L. 47
Kolk, A. J. 99
Kolthoff, I. M. 168
Kornhaas, R. 63
Kozawa, A. 24
Kriegsman, Bernard 115
Kronenberg, M. L. 42
Kruger, Jerome 34
Krupp, H. 20
Kuczynski, G. C. 187

Abstract No.

Kuper, A. B. 139
Kuttner, P. 96

LaBotz, R. J. 144
Labrie, R. J. 178
Lamb, D. M. 155
Lamb, V. A. 178
Lamoureux, R. T. 196
Lancaster, E. B. 176
Lang, Maurice 30
Larrabee, G. B. 118
Laukonis, J. V. 57
Lauriente, M. 110
Lavine, J. M. 157
Lehovec, Kurt 124
Leidheiser, Henry, Jr. 46
Levin, R. L. 51
Lewis, C. H. 150
Li, C. H. 119, 132
Linzey, Raynor 124
Litman, A. P. 35
Long, T. R. 98
Lublin, P. 148
Lurie, R. M. 28, 29
Lympany, B. B. 194
Lyons, V. J. 140

Manley, J. 106
Manz, R. C. 131, 135
Markby, Raymond 174
Marlett, C. J. 134
Mason, D. R. 144
Mathers, F. C. 77
Matovich, E. 137
Matsuda, Fujio 92
McJones, R. 20
McKinsey, C. R. 182
McNamara, J. E. 136
Meibuhr, Stuart 88
Meissner, H. P. 21
Milford, R. P. 35
Miller, J. S. 152
Miller, K. J. 131
Misch, R. D. 53
Moest, R. R. 143
Moser, J. R. 15

Nadler, Harry 199
Newman, P. A. 134
Nicholson, B. J. 198
Norman, H. 149

Oberly, J. J. 123
Odo, Keijiro 162
Ong, J. N., Jr. 54
Ord, J. L. 65
Oster, E. A. 26

Panzer, R. E. 2
Paulos, V. E. 152

Detroit Meeting 93

Philip, R. H. 173
Pick, H. J. 71
Pierson, W. W. 164, 165
Popat, P. V. 23
Porter, J. L. 155
Poster, A. R. 184
Pryor, M. J. 58
Pugh, J. W. 186

Quinn, H. F. 105, 106

Rabenhorst, H. 20
Ramaswamy, R. 158, 159
Rand, M. J. 126
Rao, K. V. 24
Read, H. J. 73, 74
Reddy, T. B. 168
Reinhardt, G. 184A
Richardson, W. W. 100
Rickel, A. M. 113
Riley, B. J. 91
Rindner, W. 157
Robertson, H. M. 136
Robinson, W. M. 129
Ruehrwein, R. A. 141
Ruka, R. 44
Russell, G. 149

Salcedo y Gumucio, Ricardo 30
Salinger, Rudolf 170
Salkind, A. J. 8
Sallo, J. S. 103, 111
Sama, D. A. 21
Sandler, Y. L. 43
Sandstede, G. 20
Sarace, J. C. 116
Scaringella, D. 132
Schindler, H. C. 127
Schmid, G. M. 68
Schumacher, E. A. 31
Schuman, Ralph 29
Scott, D. B. 49
Seifert, J. R. 153
Seiger, H. N. 10
Serravalle, G. 84
Sewell, P. B. 64
Shair, R. C. 10
Shaw, Manuel 17
Sheff, Sumner 122
Shepherd, C. M. 14
Shepherd, W. H. 156
Shimaoka, Goro 67
Shupp, B. R. 143
Sibert, M. E. 195
Silvestri, V. J. 140
Simon, A. C. 11, 12
Simonyan, Karabet 115
Smith, S. W. 39
Smith, W. K. 33
Snyder, R. N. 41

Sohns, V. E. 176
Spanos, J. 130
Spindler, W. C. 2
Steigerwald, R. F. 61
Steinitz, Robert 185
Stern, K. H. 34
Stern, M. 50
Sternberg, H. W. 174
Stewart, E. W. 151
Straumanis, M. E. 56
Strauss, Irving 185
Subcasky, W. J. 17
Sugino, Kiichiro 162
Sulich, M. 106
Swann, Sherlock, Jr. 175
Swenson, J. I. 111

Tecotzky, M. 197
Thomas, C. O. 135
Trivich, Dan 86, 87
Truesdale, R. S. 194
Tufte, O. N. 117
Turner, D. R. 82,83
Turner, J. 156
Tvarusko, Aladar 3

Udupa, H. V. K. 158, 159

Vanderkooi, Nicholas 87
Venkatachalapathy, M. S. 158, 159
Vianello, Elio 169
Vulcan, V. 128

Wagner, J. B., Jr. 51
Walker, R. D., Jr. 33
Walter, G. 20
Wang, Pei 136A
Watelski, S. B. 138
Watson, G. R. 192
Wawzonek, S. 172
Weaver, R. D. 39
Weber, H. C. 21
Weil, R. 75
Weissbart, J. 44
Wender, Irving 174
White, H. J. 99
Wilder, D. R. 179
Williams, F. V. 141
Willmann, N. L. 39
Wilson, C. L. 160, 161
Winand, René 177
Wolf, I. W. 97
Wulff, J. 181
Wynn, J. E. 25

Yeager, Ernest 24, 88
Young, G. J. 15
Youngdahl, C. A. 48

Zalabak, Charles 191
Zentner, V. 102

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